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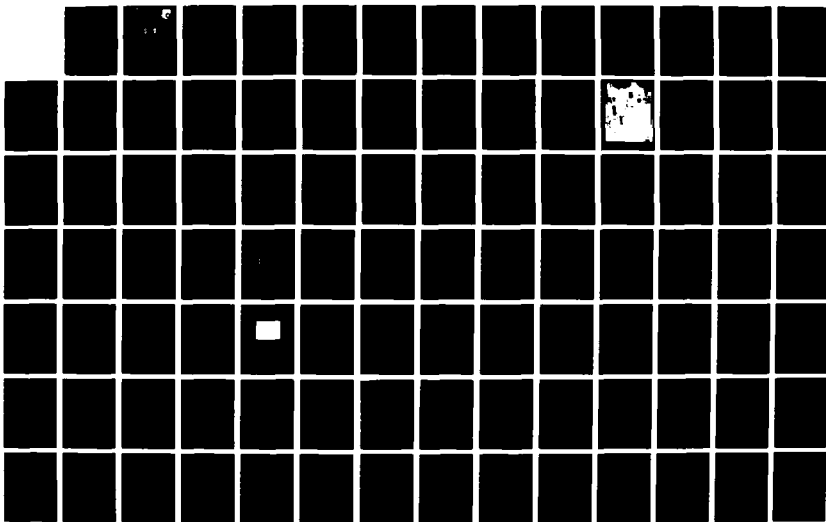
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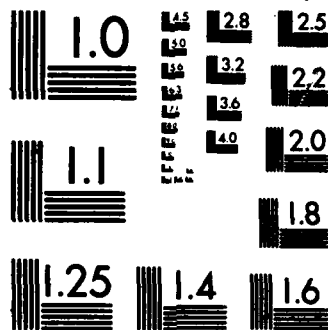
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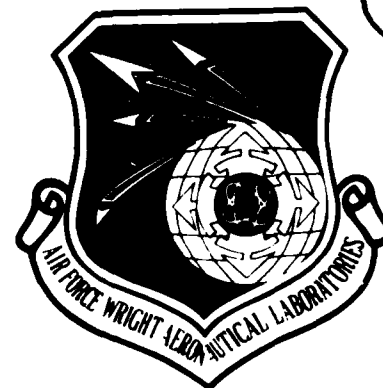


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SPACE STABLE THERMAL CONTROL COATINGS

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) An important aspect of satellite operation in a space environment is thermal control design. Various coatings having desired optical properties have been used to achieve passive thermal control of different spacecraft. IITRI's S13G/L0 coating has found widespread use in a number of missions for 15 years. The source of binder material for S13G/L0, however, is now unavailable and there is a continuing need on various spacecraft missions for this type of coating. This report covers research to develop and qualify a material having the same or improved optical and physical properties as S13G/L0. The coating was to display desirable and reliable behavior in a space environment. The study has resulted in a material designated, S13G/L0-1, which exhibits properties as good as, or somewhat better than the original S13G/L0. (Keywords: )					
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The new coating has an initial solar absorptance ( $\alpha_s$ ) of 0.18 at a thickness of 8 mils, a total normal emittance ( $\epsilon$ ) of  $0.90 \pm 0.04$ , and a  $\Delta\alpha_s$  of  $\leq 0.005$  after 1000 equivalent sun hours (ESH).

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## 1. INTRODUCTION

The objective of this program was the engineering development and qualification of a spacecraft coating having optical and physical properties equal to or better than S13G/L0. The coating would use a potassium silicate encapsulated zinc oxide pigment, be low outgassing and space stable. The investigation addressed meeting all of the requirements for a thermal control coating system(s) for space application as referenced in NASA-MSFC Specification No. 10M01835, Paint, S13G, Temperature Control, Specification for, Specification NASA MSFC Specification 10M01836, Paint S13G, Temperature Control, Application of, Specification for.

The identification and qualification of the replacement binder for the S13G/L0 thermal control coating, consisted of four major phases:

Phase 1 - Material Selection. This phase consisted of selecting and procuring the candidate silicone resin materials. Potential candidates were screened by a critical examination of the literature and by technical discussions with resin manufacturers. A minimum of two candidate materials were selected for use in the subsequent phases.

Phase 2 - Material Evaluation. The selected candidate resins were evaluated by the following studies:

1. Vacuum distillation
2. Solvent system and curing system
3. Paint formulation
4. Physical properties
5. Optical properties

Phase 3 - Space Simulation Studies. The space stability of unpigmented and pigmented candidate coatings developed in Phase 2 were determined in an in-situ irradiation facility.

Phase 4 - Manufacturing Procedures. Procedures for the manufacture of the optimized thermal control coatings were developed. Scale-up specifications, and test methods were clearly delineated.

The studies conducted on this program have resulted in the development of a new spacecraft thermal control coating designated S13G/L0-1, which has optical, physical, and mechanical properties as good as if not better than its predecessor, S13G/L0. The new coating has an initial solar absorptance ( $\alpha_s$ ) of 0.18 at a thickness of 8 mils, a total normal emittance ( $\epsilon$ ) of  $0.90 \pm 0.04$ , and a  $\Delta\alpha_s$  of  $\leq 0.005$  after 1000 equivalent sun hours (ESH).

The S13G/L0-1 has been evaluated by the University of Dayton Research Institute under contract to AFWAL/ML and by NASA/Marshall Space Flight Center. The latter organization has prepared a specification, "S13G/L0-1 Thermal Control Coating, Application of", MSFC-PROC-1384 dated 9/4/86. This document appears in Appendix A.

The S13G/L0-1 is being distributed to interested firms and organizations for their evaluation. The results of these studies will be compiled, and these data will be made available under the guidance of the Air Force.

## 2. TECHNICAL BACKGROUND

### 2.1. INTRODUCTION

The temperature regulation of satellites and space vehicles is one of the most challenging technical problems confronting spacecraft designers and engineers. The ultimate objective of thermal design is to ensure that the spacecraft operates within the prescribed temperature range defined by the temperature limitations of the vehicle materials and components. The use of selectively-reflecting surface coatings as an integral part of passive thermal design has received the most attention of the techniques available.

Overall thermal control is achieved by regulation of absorbed solar energy, solar absorptance, ( $\alpha_s$ ) and thermal emittance, ( $\epsilon$ ). The ratio of these optical properties,  $\alpha_s/\epsilon$ , is the primary materials factor in spacecraft temperature control. The thermal control coating, S13G/L0, is a well established and accepted low  $\alpha_s$  spacecraft thermal control surface coating which has been used on both manned and unmanned spacecraft. It has been utilized extensively on Air Force satellites. IIT Research Institute (IITRI) developed this coating in the 1960's<sup>1,2</sup> and is currently the sole supplier. This paint type coating utilizes a potassium silicate encapsulated zinc oxide pigment in a room temperature vulcanizing (RTV), vacuum-stripped, silicone binder (RTV-602, General Electric). It is repairable and cleanable. The selection of S13G/L0 is frequently governed by a NASA requirement that the low  $\alpha_s/\epsilon_H$  thermal control surface coating meet NASA-MFSC specification No. 10M01835. In other cases, it is selected because it has a proven flight performance record with outgassing characteristics well within the NASA-JPL criteria.

A very important fact to emerge from the recent space shuttle missions is that many materials such as Kapton, Mylar, silver, carbon, and osmium were damaged and/or removed by reaction with atomic oxygen.<sup>3</sup> In contrast, S13G/L0 was one of the few materials that showed no such atomic oxygen effects. Thus, the desirability of using S13G/L0 has been reinforced by these recent actual shuttle flight data.

## 2.2. S136/LO DEVELOPMENT

### 2.2.1. S13

Silicone paint-binders were selected for use on early U.S. spacecraft due primarily to their high-energy -Si-O-Si- backbone compared to other polymeric binders then commercially available. These early coatings were based on heat-cured polyphenylmethylsiloxane resins. In studies performed in 1959-1961, G.A. Zerlaut found that the 100% methyl silicone resins were quite superior in optical properties and space ultraviolet stability to the then used phenyl/methyl systems<sup>4</sup>. Subsequent study has not only confirmed these early results but also has shown that siloxane polymers, depending on the silicon ligand selected, are potentially the most stable candidates for use in the combined ultraviolet-plus-charged particle environment of deep space.

It was on the basis of the early experience with methyl silicone resins, (the conclusions of which were subsequently reinforced by studies at IIT Research Institute for the Jet Propulsion Laboratory) that S13 was developed.<sup>1</sup> This elastomeric silicone formulation combined the apparently stable SP500 ZnO (New Jersey Zinc) with GE's RTV 602.

Zinc oxide (ZnO) was, until 1965, thought to be the most stable white pigment available in terms of the stability of its optical properties to ultraviolet irradiation in vacuum.<sup>5</sup> However, in 1965 serious challenges to ZnO's purported behavior were reported as discrepancies arose between laboratory-simulation data and flight-experiment data obtained from the materials' experiments flown on OSO-II<sup>6</sup> and the Pegasus<sup>7</sup> spacecraft.

These data indicated that ZnO-based silicone coatings were considerably less stable than predicted by the extensive space-simulation testing to which they had been subjected. This instability has since been attributed to the formation of an easily-bleached (by oxygen) infrared absorption band that is not observed by the postexposure reflectance measurements that were at that time performed in air. This absorption band was first observed in the laboratory by MacMillan et al<sup>8</sup> during in-situ measurements of the bidirectional reflectance of in vacuo, ultraviolet-irradiated ZnO.

Figure 1 shows the effect of ultraviolet irradiation on IITRI's S-13 thermal control coating (the polydimethylsiloxane, General Electric RTV-602, paint pigmented with untreated ZnO) that had degraded severely in flight tests<sup>6,7</sup>. The S-13 paint exhibits a reflectance decrease of about 35% at 2000 nm ( $2\mu$ ) after approximately 800 equivalent sun hours (ESH) of ultraviolet irradiation in vacuum. However, an essentially instantaneous increase in infrared reflectance occurs when either air or oxygen is admitted to the irradiated specimen; the recovery is very nearly total after only two minutes of exposure to air (Figure 1).

The effect of 1000 ESH of ultraviolet radiation on a pigment specimen of SP500 ZnO is shown in Figure 2. These data show that the bulk of the solar absorptance increase exhibited by ZnO as a powder, and as a paint in the S-13 coating, is due to the oxygen-bleaching damage sustained in the infrared.

The rapidity that the ultraviolet-induced infrared absorption band develops in ZnO and the abruptness with which oxygen annihilates the absorption strongly indicates that the infrared phenomenon is not related to bulk diffusion phenomena but is associated with the photodesorption of absorbed oxygen. IITRI's Z93 thermal control coating, an SP500 ZnO-pigmented potassium silicate paint, did not undergo the bleachable infrared damage exhibited by S-13 and pure ZnO. Figure 3 is a typical spectra of irradiated and unirradiated Z93, which is seen to be quite stable to ultraviolet radiation in vacuum.

#### 2.2.2. S13G

The absence of damage to ZnO-pigmented alkali silicate coatings suggested that the reaction of ZnO with the potassium silicate may have precluded the bleachable infrared degradation exhibited by ZnO powder and ZnO-pigmented silicone paints. The polydimethylsiloxanes do not "wet" the pigment particles and consequently it was conjectured that they do not offer an effective barrier to photodesorption reactions on the surface of ZnO. A series of experiments were performed in which ZnO was first reacted with potassium silicate and then extracted and dried as a new, treated ZnO pigment. It was shown that a ZnO powder treated (reactively encapsulated) in this way does not exhibit infrared degradation. The reflectance spectra of an irradiated silicate-treated ZnO-specimen are presented in Figure 4.

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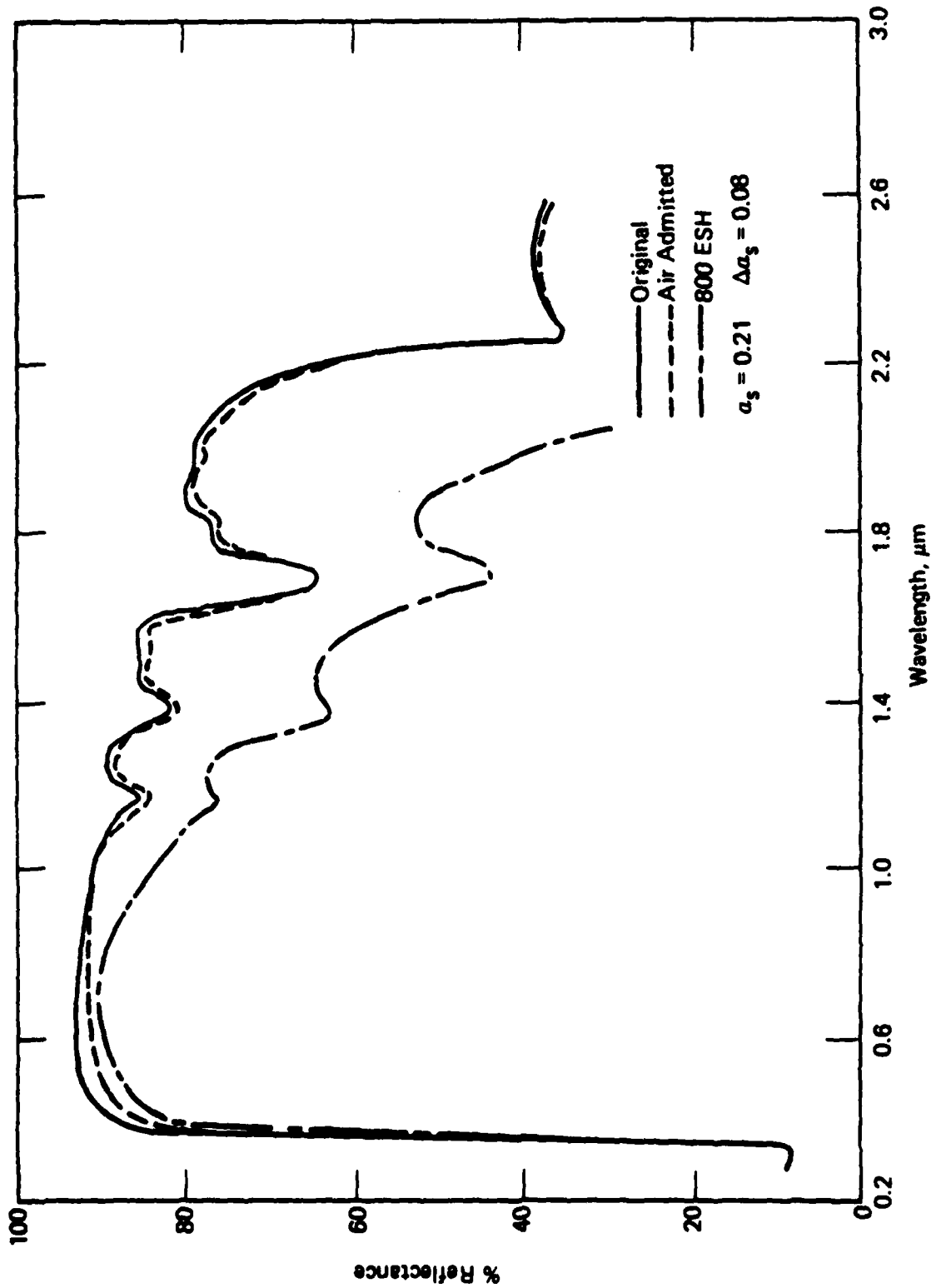


Figure 1. UV Degradation of S-13.

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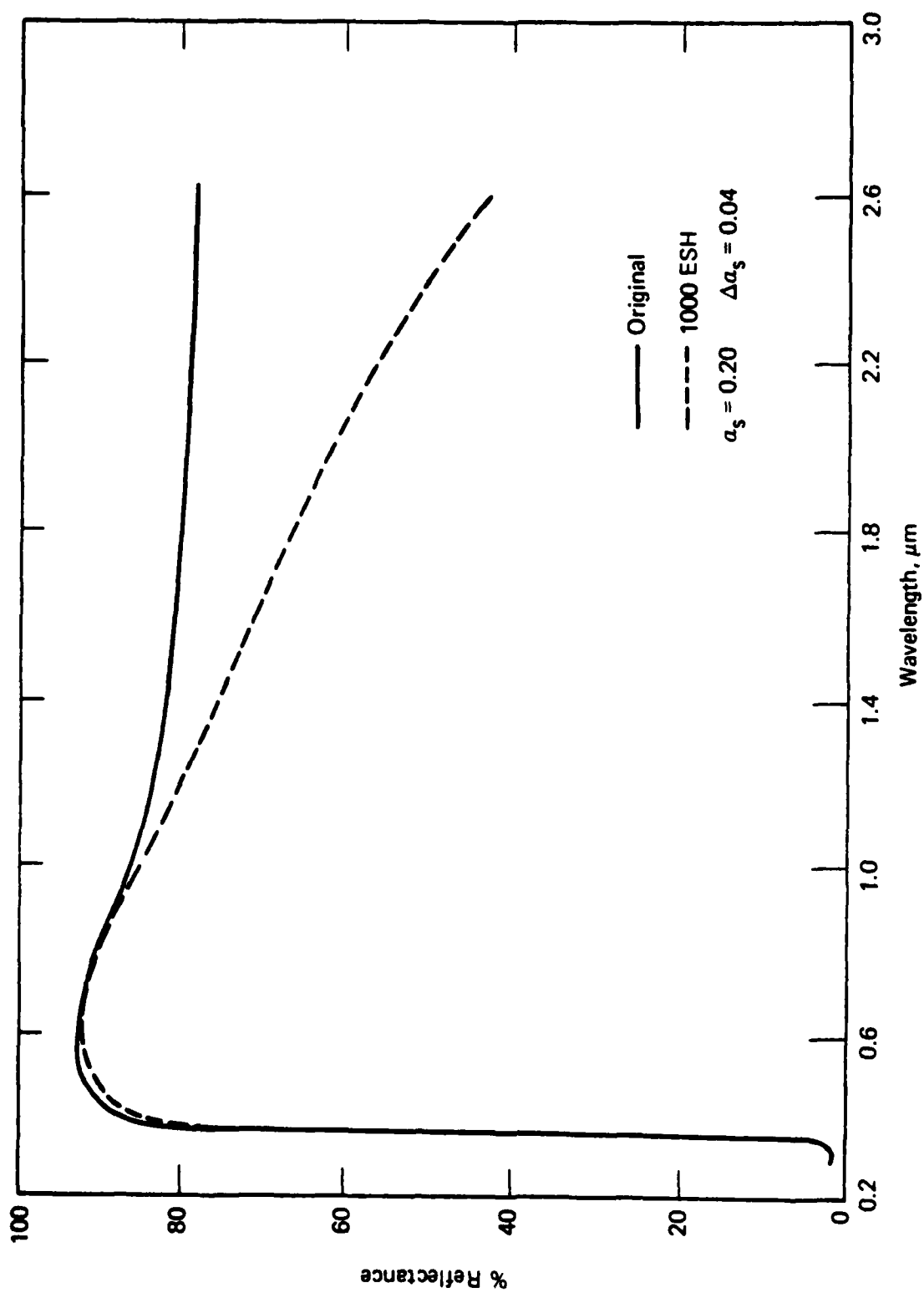


Figure 2. UV Degradation of ZnO

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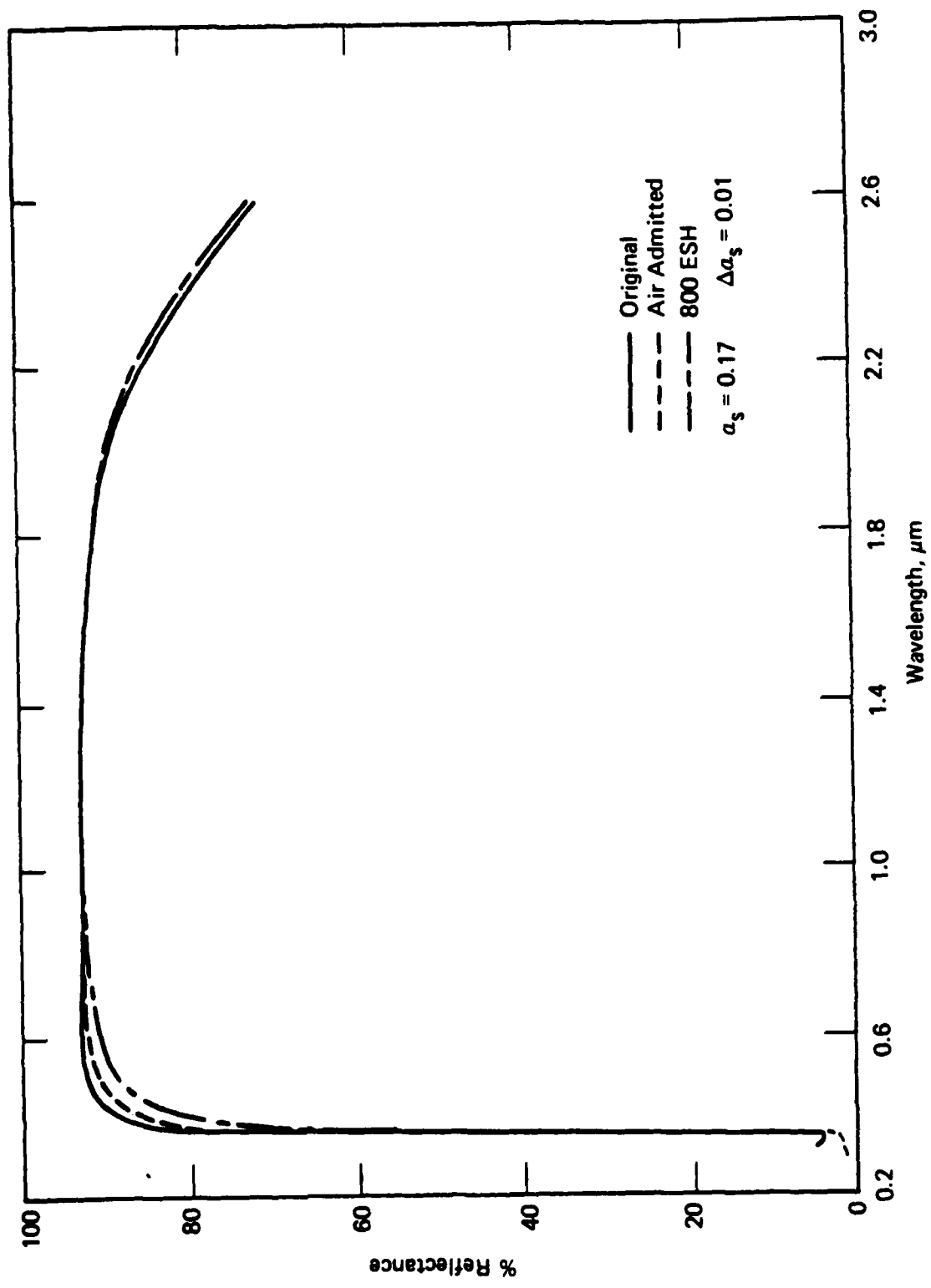


Figure 3. UV Degradation of Z93.

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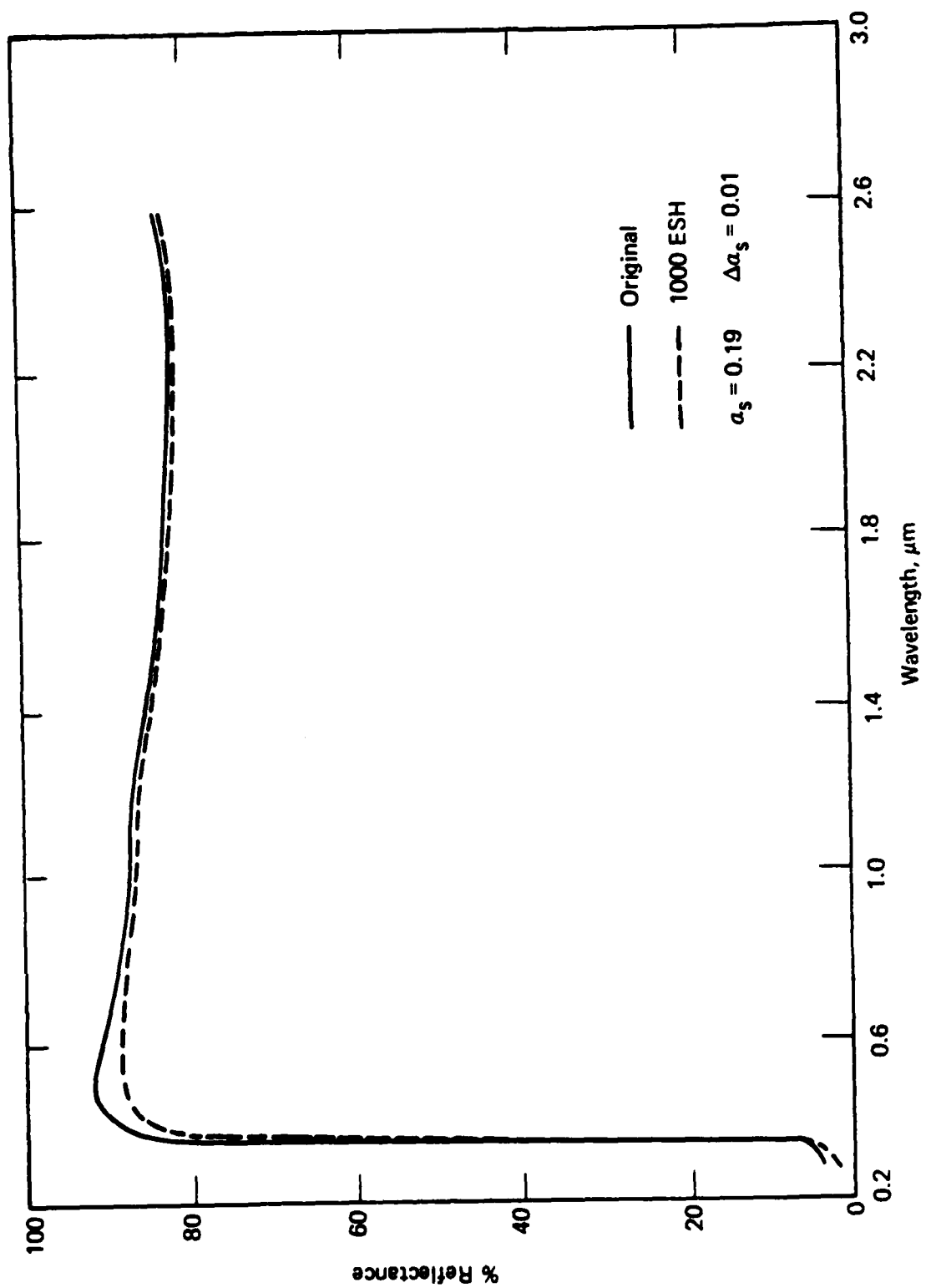


Figure 4. UV Degradation of Silicate-Treated ZnO

The infrared optical behavior of ZnO may be explained on the basis of a free-carrier absorption mechanism. Gilligan<sup>9</sup> in studies conducted for the Jet Propulsion Laboratory, concluded that the absorbed photon creates an electron-hole pair with the hole migrating to the surface to discharge absorbed oxygen species. The valence-band holes are attracted to the surface, where they discharge absorbed oxygen (and other absorbate gases). The surface thus becomes electron rich with the result being that the electrons accumulate in the infrared-active conduction band where, if their concentration is high enough, it is observed as the so-called free-carrier absorption.

Obviously silicating with potassium silicate interferes with the kinetics of electron accumulation in the conduction band. It may be postulated that the polynegativity of the silicate anion attracts the "positive" holes preferentially with the result that the electrons are no longer as free to accumulate in the conduction band.

The S-13G paint was formulated with the reactively encapsulated ZnO to replace the S-13 coating which had been specified for a large number of spacecraft. The elastomeric S-13G paint was desired for those applications where the use of Z93 was precluded or presented difficulties. For example, the silicone coating is more easily applied, can be manufactured and shipped (as opposed to Z93) and is, unlike Z93, easily cleaned.

It was realized during the course of the early studies that the development of stabilized ZnO-pigmented RTV-602 silicone paints was largely a process problem. Factors that had to be considered in engineering S-13G were <sup>10,11</sup>:

1. Initial ZnO-silicate reaction parameters such as materials' balance, reaction temperature, reaction time, and mixing during reaction.
2. Pigment filtration and silicate extraction (washing) procedures.
3. Pigment-drying and -grinding procedures.
4. Silicone paint-manufacturing procedures.
5. Optimization of paint formula relative to pigment volume concentration (PVC), solvents employed, catalyst concentration, etc.

### 2.2.3. S13G/L0

The deleterious effect of outgassing, i.e., volatilization of degradable components from spacecraft surfaces which would condense on critical areas such as optics, was recognized in the early 70's. Criteria were established by NASA for an outgassing test involving exposure of the test material for 24 hours to 125°C at a pressure of less than  $10^{-6}$  torr. The sample is weighed before and after the test to determine total weight loss (TWL). The volatile condensable material (VCM) is determined from the amount of material condensing on a collector plate maintained at 25°C.

It was determined that the standard production batch S13G was marginal in meeting the 1.0% TWL and 0.1% VCM. Studies were conducted to develop improved stripping procedures for the RTV 602. Molecular distillation to remove low molecular weight components was found to be an effective means of obtaining the desired minimal outgassing characteristics<sup>12</sup>. The result of these studies was S13G/L0, the recent generation elastomeric thermal control coating formulation.

### 3. RESULTS AND DISCUSSION

#### 3.1. MATERIALS SELECTION AND ACQUISITION

The identification of resins which would be viable candidates for S13G/L0-1 was the primary activity in the initial phases of this program. The requirement was for a silicone resin with characteristics similar to those of RTV 602. The initial criteria were curing behavior at room temperature, clarity and purity, elastomeric properties in the cured state, and availability. The last-mentioned factor was an important consideration to assure continuity in obtaining an S13G/L0 type of coating.

A list of candidate commercial silicone resins is presented in Table 1. As shown, the four major suppliers of silicone resins whose products were included for evaluation were: SWS Silicones Corporation in Ardrian, Michigan; McGhan NuSil Corporation in Carpinteria, California; General Electric Co. in Waterford, New York; and Dow Corning Corporation of Midland, Michigan. The choice of candidates was also made with the aid of suggestions and information supplied by these manufacturers and also by colleagues in aerospace and associated companies and different government agencies.

#### 3.2. RESIN STUDIES

During the course of this program, resin studies and pigmented resin studies were conducted in parallel, benefitting from "cross-feeding" of data. For clarity in this report, these two areas will be discussed in separate sections.

##### 3.2.1. Vacuum Distillation

In order to render silicone resins non-contaminating in a space environment, the low molecular weight fractions must be removed through distillation. Studies were conducted in a molecular still to determine stripping behavior under various conditions and the effects on resin properties.

TABLE 1. CANDIDATE COMMERCIAL SILICONE RESINS

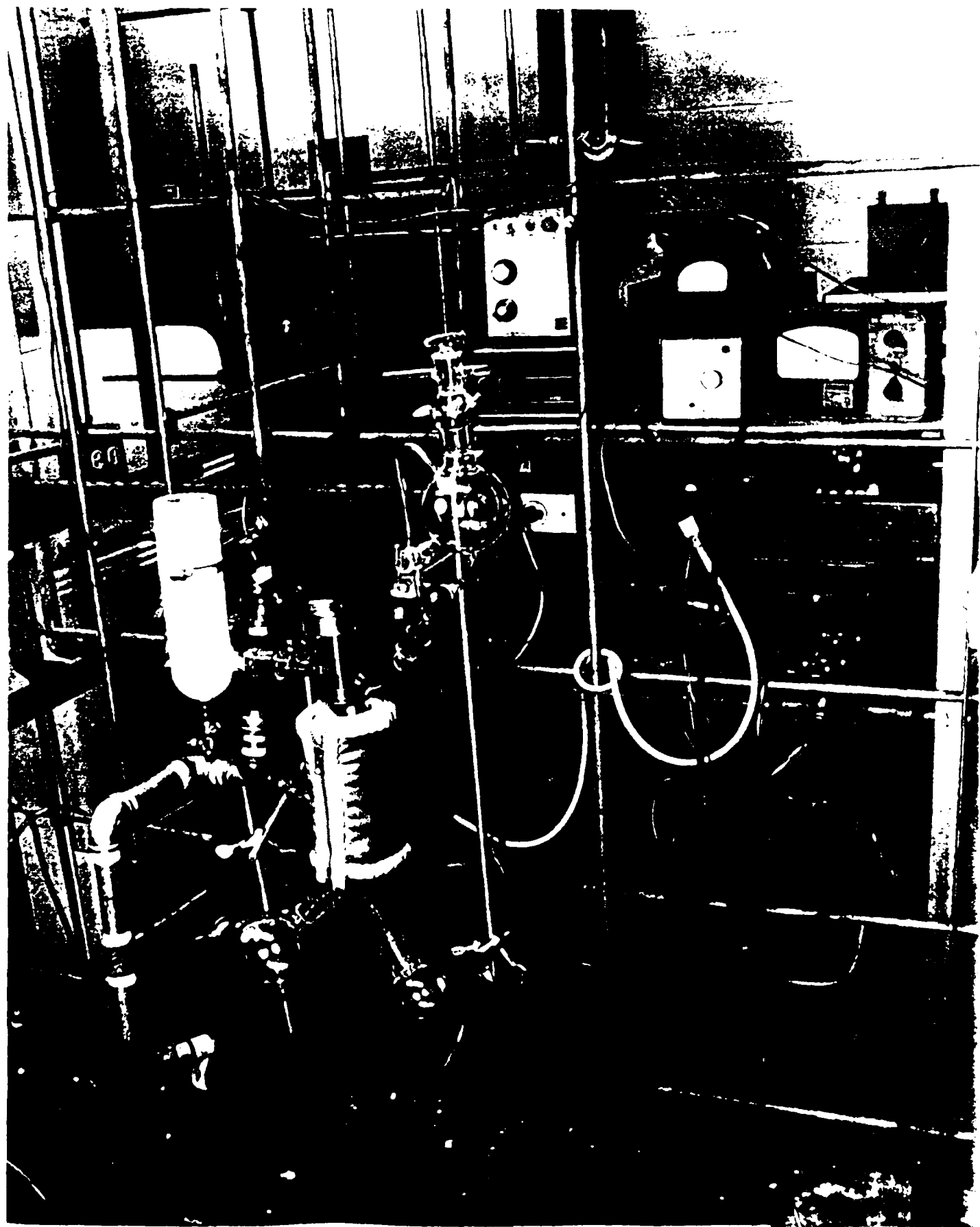
Manufacturer	Material Designation	Description
SWS-Silicones Corp. (Adrian, MI)	SWS-810	RTV silicone rubber kit, transparent
	V-81	RTV silicone rubber resin kit, transparent
	V-10	RTV silicone rubber resin kit, transparent
McGhan NuSil Corp. (Carpinteria, CA)	R-2500	Controlled volatility RTV silicone (clear) kit
	R-2567	Controlled volatility RTV silicone (clear) kit
	R-2602	RTV silicone
	R-2615	RTV silicone elastomer kit (clear)
	R-1009	RTV silicone dispersion coating (translucent)
General Electric Co. (Waterford, NY)	RTV-615	RTV silicone resin kit (clear)
	RTV-655	RTV silicone resin kit (clear)
	RTV-670	RTV silicone resin kit (clear)
	RTV-12	RTV silicone resin kit (clear)
Dow Corning Corp. (Midland, MI)	R-6103	Two component flexible silicone coating kit (clear)
	93-500	Dow space qualified resin kit
	Sylgard 186	RTV silicone resin kit (clear)
	Sylgard 184	RTV silicone resin kit (clear)
	Sylgard 182	RTV silicone resin kit (clear)
	DC-3140	RTV silicone (adhesive/sealant) rubber (translucent)
	3-6527	RTV silicone non-corrosive dielectric gel (clear)
	DC-2577	One component silicone conform coating (translucent)

The vacuum distillation equipment used is shown in Figure 5. The system consists of:

1. Resin feed flask equipped with a teflon flow valve.
2. Vacuum distillation chamber with water cooled central condenser, resin receiver flask, distillate receiver flask, and vacuum thermocouple.
3. Motor driven and electronically controlled rotary vacuum feedthrough. The rotary feedthrough is connected to film wiper arms equipped with teflon film wipers.
4. Liquid nitrogen cold trap.
5. 52 cfm mechanical two stage pump, which provides a maximum vacuum of  $1 \times 10^{-4}$  torr.

The procedure employed when distilling an uncharacterized resin is as follows:

1. The still is taken apart and carefully cleaned to prevent reaction of any compound that may be present in the still with the resin under study. This is done to insure that gelation does not occur, which can cause overheating, seizure, and fracture of the glass distillation chamber. Further, it significantly reduces the possibility that discoloration will occur. The still is then air dried for about 1/2 hr and reassembled.
2. Once assembled and all joints are determined to be leak tight, the system is evacuated and vacuum dried for several minutes until the system vacuum gauge indicates approximately 5 $\mu$  pressure. The cold water for the central condenser is also turned on at this time.
3. LN<sub>2</sub> is filled and allowed to come to equilibrium.
4. 350-400 mls of resin are introduced into the feed flask which is sealed from the rest of the system by two closed valves, one being the teflon flow valve and the other, a bypass valve located at the top of the feed flask.
5. The feed flask is then completely sealed by a combination ground glass stopper/valve. The resin is then deaerated by slowly opening and closing the bypass valve as required.
6. The heating mantle is then positioned around the distillation chamber and turned on.
7. The film wiper is turned on and adjusted to 300-350 rpm.
8. The flow valve is then adjusted to allow a small amount of resin to flow down towards the distillation chamber. It is timed to arrive as the walls of the chamber reach operating temperature.



9. As the resin arrives at the mouth of the distillation chamber which is now at the correct temperature, the film wiper should be adjusted to 700 rpm.
10. A check is then made of the internal operating pressure and adjusted to maintain a pressure of less than 20 microns.
11. The pressure, LN<sub>2</sub> trap, and flow valve should be monitored and appropriate action taken to maintain correct parameters at least every 1/2 hr.
12. When the run is completed, the heating mantle is removed and the temperature controller shut down. The flow valve is closed and wiper rpm is reduced to 300-350 until chamber walls cool to less than 100°C.
13. Once the distillation chamber is cool, the vacuum pump is sealed and the system is brought to atmospheric pressure. The recovery flasks can then be separated from the system to remove the resin and distillate for further work.

#### 3.2.1.2. Results

The results of several distillation experiments are shown in Table 2. All of the resins produced a range of between 3 to 8 cc of distillate per 100 cc of resin, indicating removal of undesirable potentially outgassing material. These amounts are higher than that generally obtained for RTV-602 as shown in the Table. Evaluation tests also were conducted to determine if any problems in cure resulted from distillation process. No negative effects were apparent. In fact, the process may actually aid curing by the removal of inhibitory substances generally found in the material. As can be seen, most of the resins were higher in viscosity than RTV-602. Except for the Dow resins, the materials were fluid enough to have acceptable workability.

The SWS resins which initially have a straw color retain such coloring after distillation. An experiment was developed and undertaken to determine if the resin could be decolorized:

1. 20 gm of SWS-810 resin were weighed and placed in a round bottom flask.
2. 2 gm of decolorizing charcoal were added with 80 gm of toluene.
3. The mixture was then stirred to achieve a homogeneous mixture and heated in a hot water bath at 80°C for 1 hr while stirring.

TABLE 2. SILICONE DISTILLATION CONDITIONS AND RESULTS

Manu- facturer	Resin	Resin Volume, cc	Viscosity, cp		Vacuum, $\mu$	Volume of Distillate, per 100 cc resin
			Initial	Final		
Dow	182	350	9,890	10,040	6	4.6
Dow	184	350	7,800	10,580	6	5.1
SWS	V-81	350	870	1,290	35	7.1
SWS	810	300	5,300	7,200	6	6.7
GE	RTV-615	400	4,600	4,800	50	7.0
MN	R2602	400	1,940	2,300	325	6.0
GE	RTV-602	1000	--	1,200	10	3.8
GE	RTV-12	400	1,450	1,480	8	3.3
SWS	810	300	5,300	7,190	6	3.0
SWS	V-10	400	4,950	6,150	10 1st pass 2 2nd pass	5.0
SWS	V-81	400	1,030	1,520	100 1st pass 3 2nd pass	7.5
MN	R2602	400	2,720	3,960	14 1st pass 1 2nd pass	3.0

Note: Distillations conducted at 210°C

a. Average value of all readings taken during distillation

4. The mixture was then cooled to room temperature by standing and filtered using 12.5 cm filter paper, medium-fine porosity with an 8  $\mu$ m particle retention.
5. The filtrate was refiltered using a  $MgSO_4$  plug to completely remove all of the carbon.
6. The filtrate was decanted into a vacuum roto still and the solvent removed. The resulting resin was clear with no visible discoloration observable.

### **3.2.2. Volatile Condensible Materials/Total Weight Loss**

#### **3.2.2.1. Test Method**

The resins, with the exception of the controlled volatility resin R-2657, were vacuum distilled under the conditions outlined in the preceding Section. Catalyst and solvent were then added, and the mixture was deposited onto a Mylar substrate using air spraying. Spraying conditions were an air pressure of approximately 30-35 psi with an average distance to the substrate of 10-12 in. The silicone films were allowed to air dry at room temperature for 2 weeks. An adequate size sample (approximately 2 in. x 3 in.) was then removed from the substrate and submitted for testing for VCM/TWL.

The testing of the films was carried out by Ball Aerospace Systems Division according to their Method No. 33074 entitled, "Short Term Weight Loss and Condensed Contaminant Testing of Materials in a Thermal Vacuum Environment." The test is similar to ASTM E-595-77 and consists of preconditioning the sample for 24 hr at 25°C and 50% relative humidity. The test sample is then exposed to 125°C,  $10^{-5}$  torr environment for 24 hr. The collector is maintained at a temperature of 25°C for the collection of volatile condensable materials. Sample weight is determined before and after exposure to the 24 hour vacuum test.

Samples were also submitted to the McGhan Nusil Corporation for testing. Their testing was done in accordance with ASTM-E-595-77 and NASA SP-R-0022A specifications. The basic test parameters are the same as those of BASD 33074.

### 3.2.2.2. Results

Results of early VCM/TWL tests at Ball Aerospace are presented in Table 3. Examination of the data shows that all of the materials exceed the accepted NASA limits for TWL and VCM. Based on past performance of S13G/L0, one may expect a possible reduction in VCM of about 50%, and TWL may be reduced by 0.1% with incorporation of a pigment. For example, an average VCM/TWL for S13G/L0 that has not undergone post cure conditioning is 0.06/0.5. However, based on this assumption only the RTV-602 (used here as the reference standard) and SWS V81 would meet specifications.

The performance of the MN R-2567 was unexpected. It is reportedly a controlled volatility material and therefore was not subjected to vacuum distillation as were the other materials. However, its poor thermal vacuum characteristics, as shown in Table 3, indicate that this material might have had improved TWL/VCM values if it had been vacuum distilled as well.

The other resins suffer from high amounts of VCM, which would likely exceed accepted values even in pigmented form. The MN R-2602 (IITRI Batch G-087) exhibited the worst VCM of the candidate resins, but this is probably due to the high pressure under which it was initially processed (325 $\mu$ ) relative to the other resins. The SWS V81 and SW 810 were processed at pressures of 36 and 6 $\mu$ , respectively. This was substantially lower than that for R-2602 and may account for R-2602's higher VCM/TWL values. Further work was conducted in optimizing distillation parameters, which had a favorable effect on material VCM/TWL characteristics. These improvements were upgrading of the vacuum capabilities/instrumentation and maintaining a 220°C processing temperature.

Parallel studies with pigmented resin formulations indicated some difference in test results from the different laboratories. Identical samples were submitted to both Ball Aerospace and McGhan NuSil for evaluation. Each sample set consisted of one sample of RTV-602 and RTV-12, both of which had been stripped. The RTV-602 was cured using 10% TMG (tetramethylguanadine) and RTV-12 with the GE-supplied catalysts, which had a blue color for identification and traceability. After the catalyst was added and mixed, two samples of each material were made and allowed to cure for ten days under identical conditions. One sample set of each material was identically

TABLE 3. VCM/TWL ANALYSIS OF CURED SILICONE RESINS

<u>Mfr.</u>	<u>Material</u>	<u>Weight Loss, %</u>	<u>VCM, %</u>	<u>Forward Light Shutter, %/cm<sup>2</sup></u>	<u>Visible VCM Deposit</u>
SWS	V81	1.094	0.173	$7.45 \times 10^{-2}$	+
SWS	810	1.673	0.474	0.251	+
MN	R2602	2.5	0.692	0.43	+
MN	R2567	2.028	0.667	1.591	+
GE	RTV-602/L0	0.472	0.157	$6.39 \times 10^{-2}$	+

prepared, and a set was sent to each laboratory for testing. The only difference between samples was the identification code number.

As can be seen from the data in Table 4, the VCM/TWL results differed appreciably. It is unknown whether this variance reflects differences in handling, in test systems and parameters, and/or a combination of these variables. In the case of both resins, however, the VCM/TWL results are well within the NASA criteria of 0.1%/1.0%. Copies of data sheets from the two laboratories are presented as Appendix B to this report.

TABLE 4. TML/VCM RESULTS

Designation	McGhan NuSil		Ball Aerospace Co.	
	VCM, %	TML, %	VCM, %	TML, %
RTV-602	0.00	0.28	0.027	0.406
RTV-12	0.01	0.12	0.034	0.242

### 3.2.3. Optical Characterization

#### 3.2.3.1. Methods

Several methods of resin sample preparation were evaluated for use in both IR and UV-VIS-NIR material characterization. The first method employed the direct deposition of catalyzed resin to the appropriate substrate, i.e., quartz for 190-2600  $\mu\text{m}$  region and KBr for the 2.5-30.0  $\mu\text{m}$  region, using solvent diluted solutions of resins. This method was found to be unsatisfactory for the following reasons:

1. Formation of scattering centers caused by trapped air bubbles.
2. Meniscus formation resulting in nonuniform coating thickness.
3. Excessive coating thickness for IR measurements.
4. Resin cure inhibition caused by solvents or water absorbed by the solvents.

A second method consisted of casting 100% resin onto the substrate with a 0.05 in. spacer frame, and then removing any excess by running the flat edge of a microscope slide across the surface of the spacer. The resin was then allowed to cure for 24 hr. This method generally worked well, but in some

cases still resulted in the formation of a meniscus. The resulting curvature can cause substantial displacement of the spectrum during detector or source changes.

The third method was essentially the same as the second, but a cover plate made of the same material as the substrate was carefully lowered onto the surface. This is done by placing the cover plate at about a 70° angle relative to the plane of the substrate and decreasing the angle until the coverplate completely rests on the spacer. Care must be taken to insure that no air entrapment occurs during the process. The resin is then allowed to cure for 24 hrs. This was found to be the best method since it eliminated the lensing effect from meniscus formation which cause detector and source change displacement problems. The samples were free from air bubbles that cause scattering, and they were of a relatively uniform thickness.

#### **3.2.3.2. Instrumentation**

All UV-VIS-NIR transmittance measurements were made using a Perkin-Elmer SF-340 spectrophotometer. The instrument has a range of 190-2600 nm and the transmittance measurements are done in the normal-normal  $\tau_{NN}$  operating mode. The scale used was 1/2x with a scan time of 12 min.

Mid IR measurements were made using a Perkin-Elmer 580B spectrophotometer, also in normal-normal transmittance mode of operation. This instrument has a range of 2.5-50  $\mu\text{m}$  and all measurements were made using a 1X scale with a scan time of 8 min. The reflectance measurements to determine solar absorptance utilized a Beckman DK-2A spectrophotometer with an Edward's type absolute integrating sphere. Its mode of operation is normal hemispherical reflectance ( $p_{NH}$ ), with a scan time of 12 min. It is capable of doing absolute normal-normal  $\tau_{NN}$  and normal-hemispherical transmittance  $\tau_{NH}$ . The SF340 was utilized for transmittance measurements because of its greater resolution capability.

#### **3.2.3.3. Characterization**

Figures 6-10 are infrared spectra of R2602, SWS810, V81, R2567, and RTV-602, respectively. Although the spectra are somewhat obscured by excessive sample thickness, some differences in signature can be seen. The primary difference in the spectra is the aromatic C-H absorption at about 3006  $\text{cm}^{-1}$

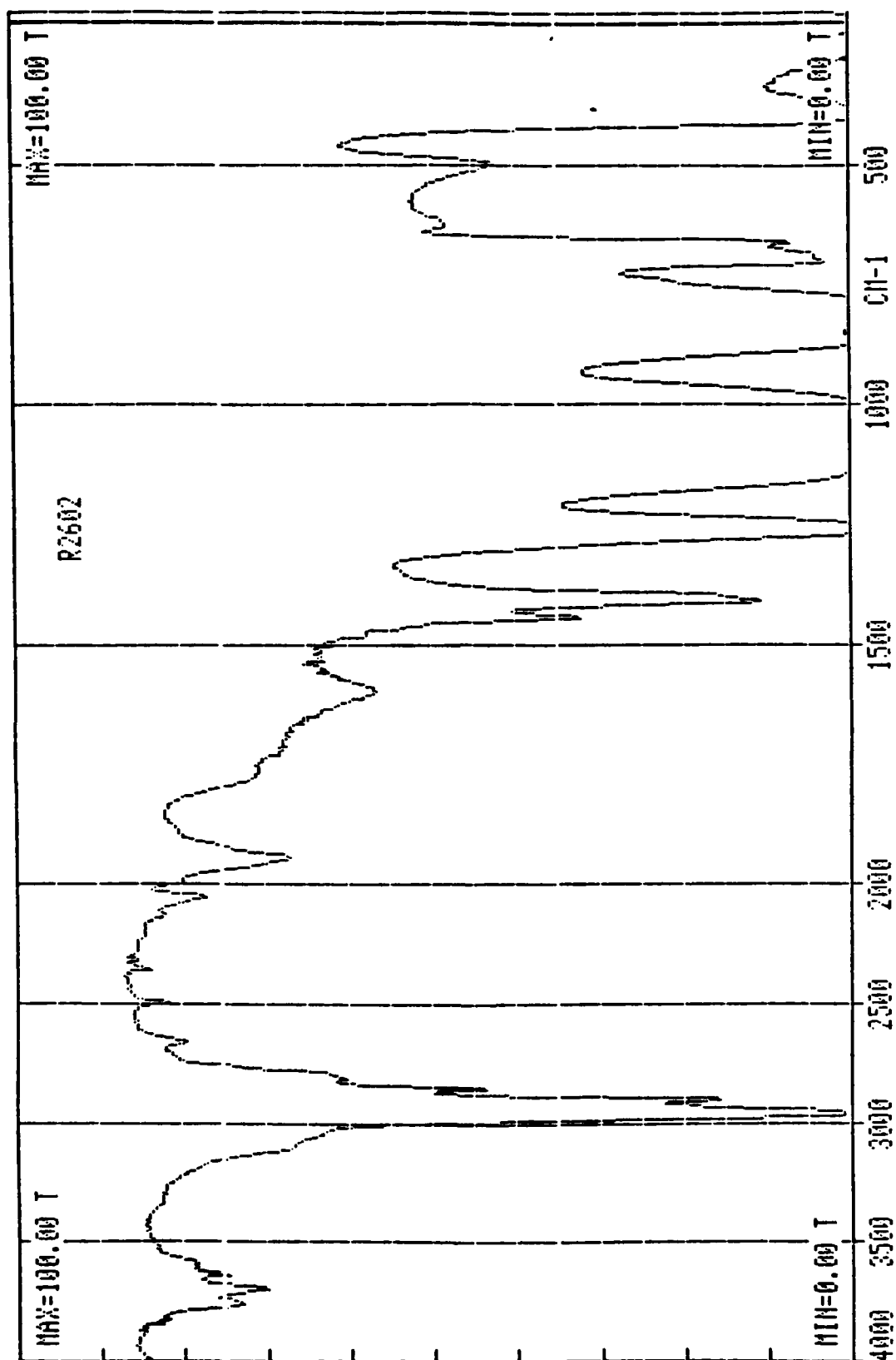


Figure 6. Normal-normal IR transmittance spectrum of McGhan Nusil R2602 cured resin.

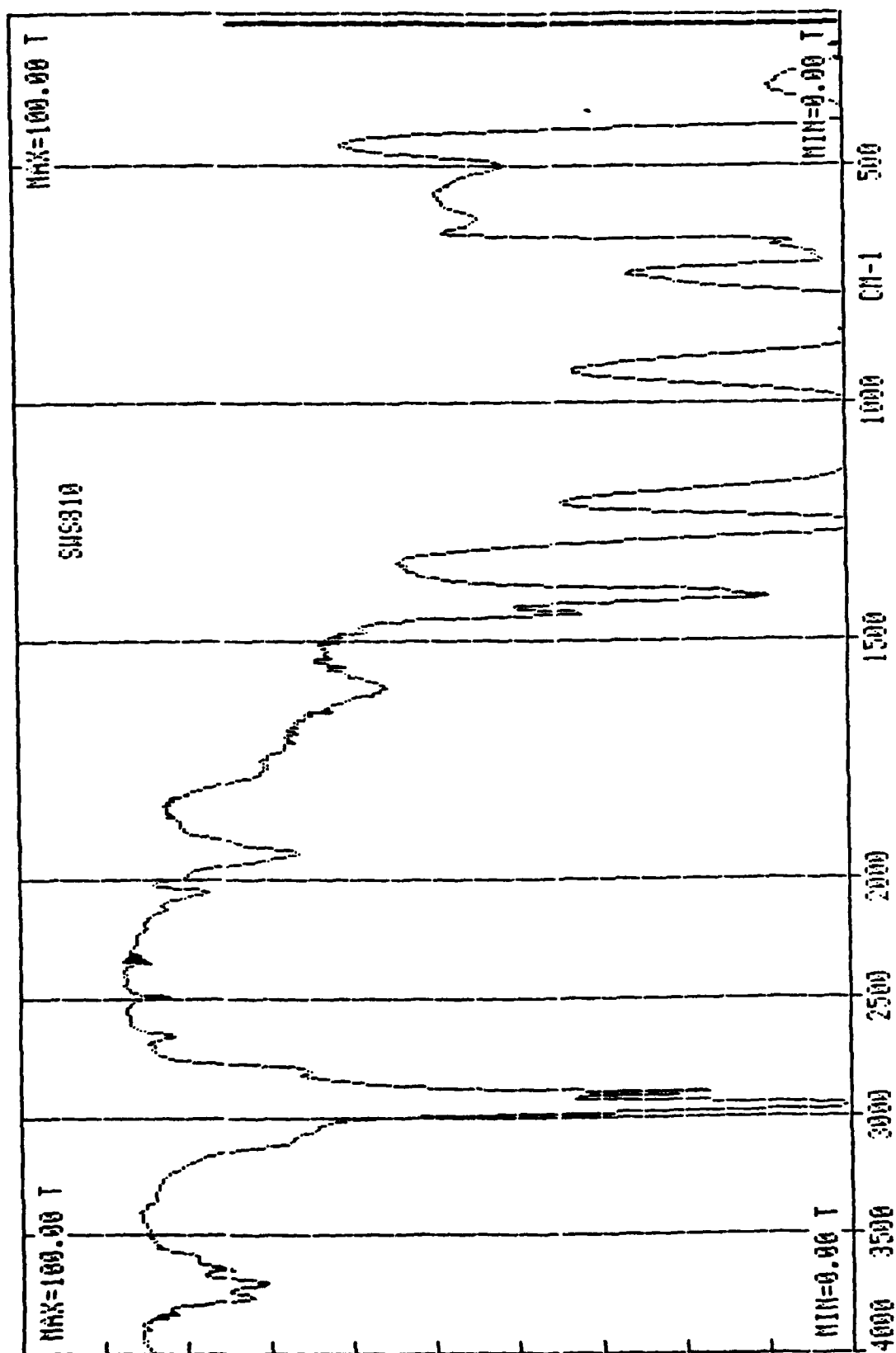


Figure 7. Normal-normal IR transmittance spectrum of SWS-810 cured resin.

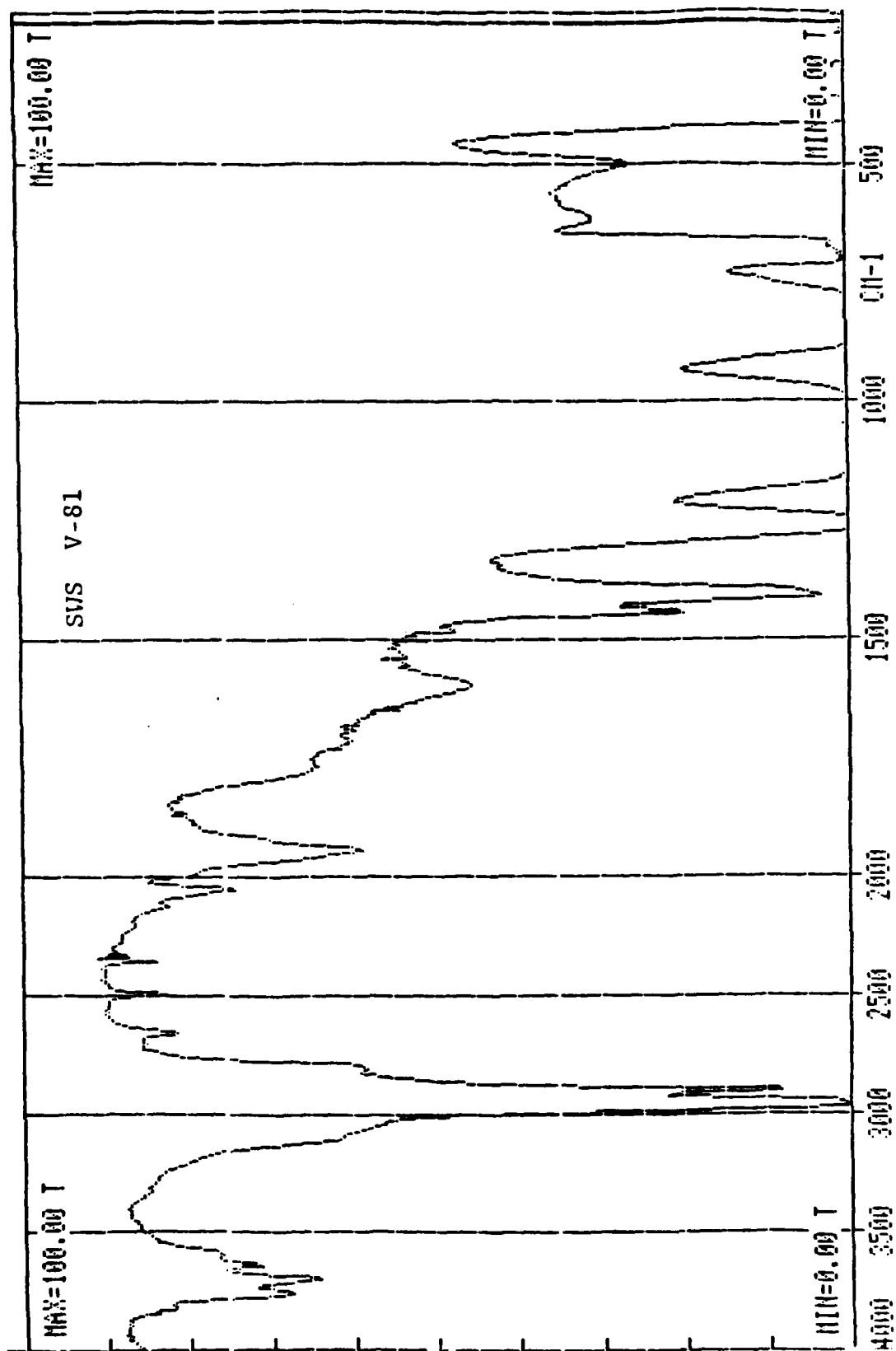


Figure 8. Normal-normal IR transmittance spectrum of SWS V-81 cured resin.

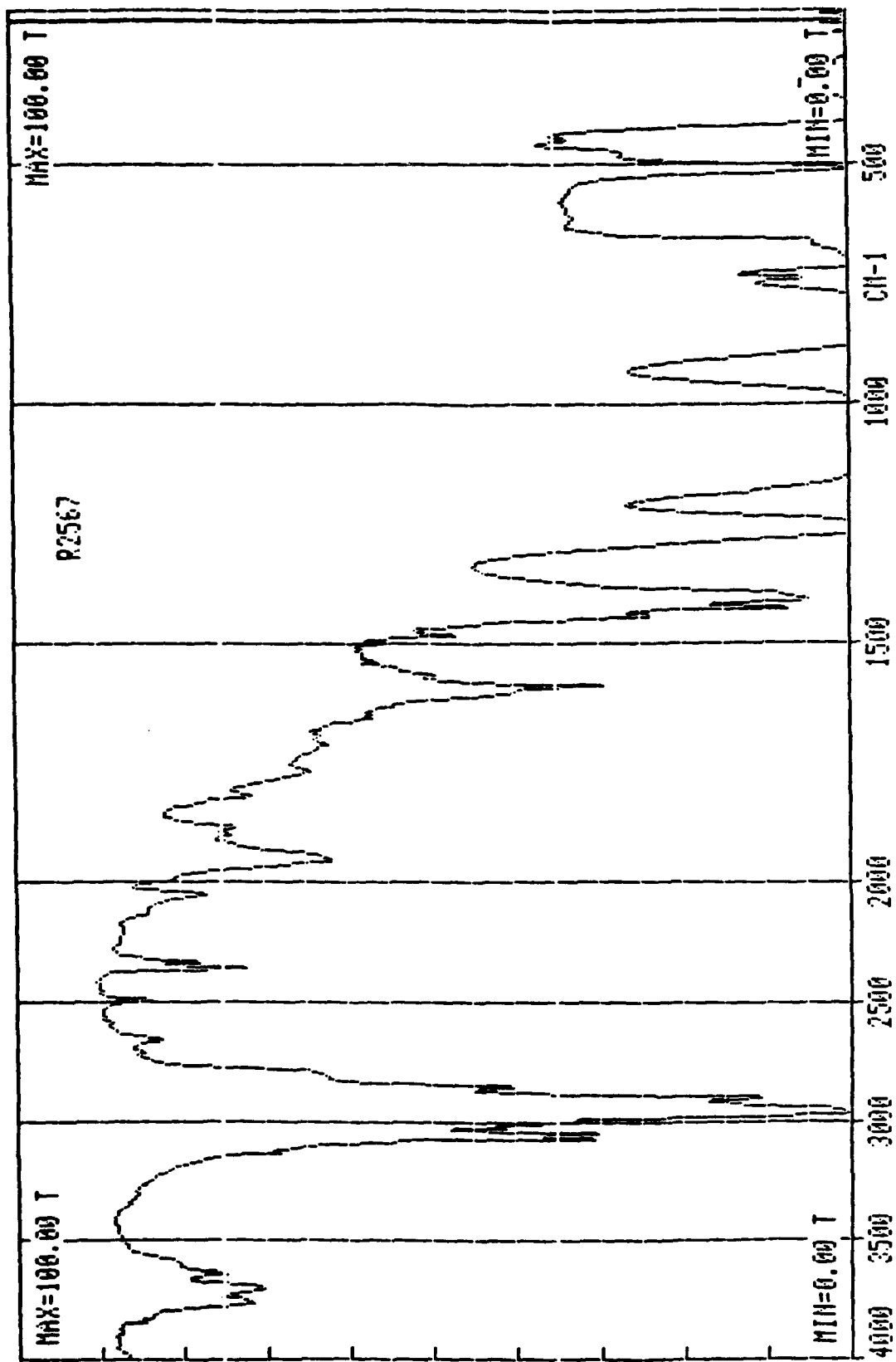


Figure 9. Normal-normal IR transmittance spectrum of McGhan Nus11 R2567 cured resin.

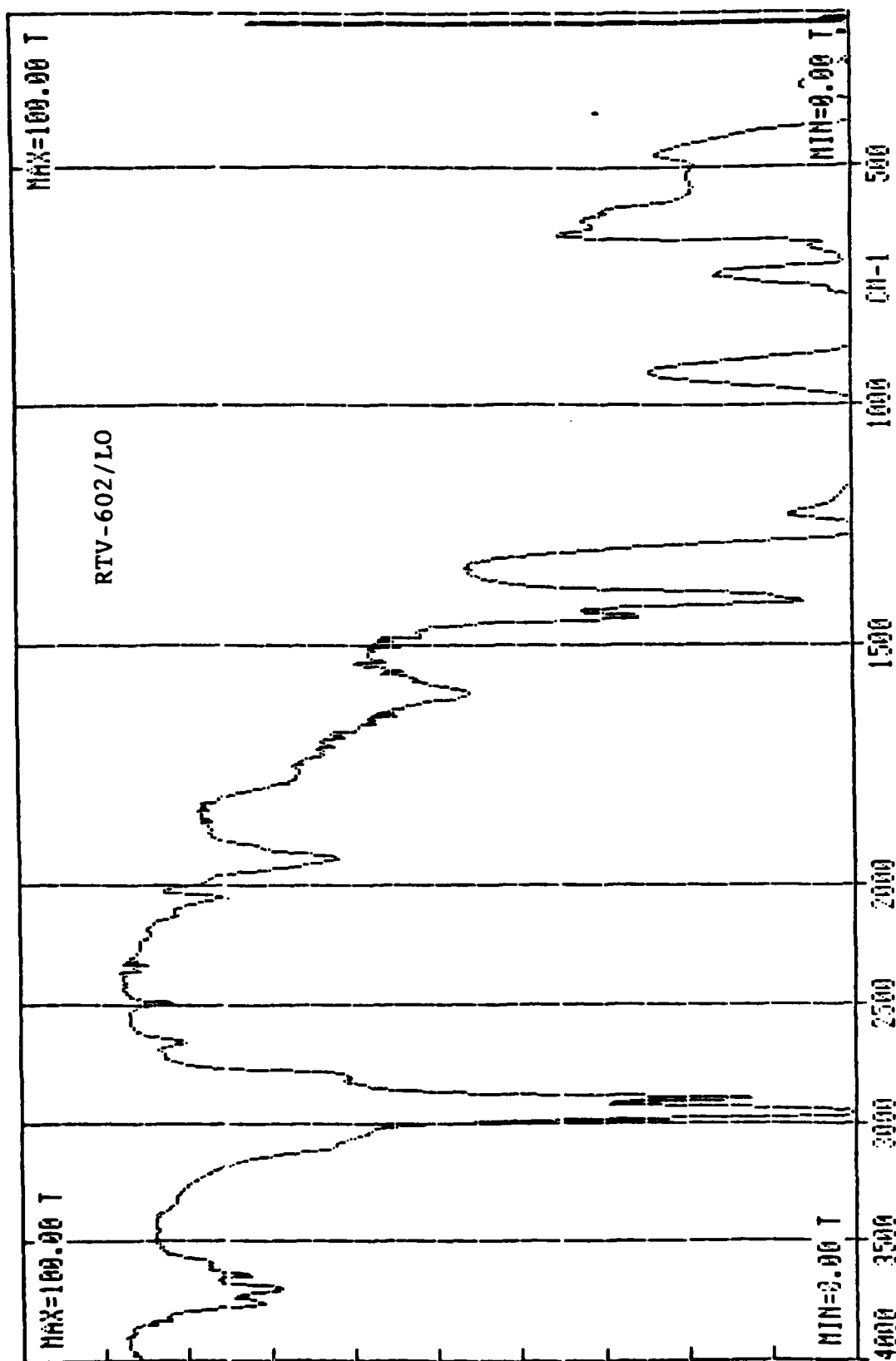


Figure 10. Normal-normal IR transmittance spectrum of GE RTV-602/L0 cured resin.

for R2567 shown in Figure 9, and the sharp absorption band at about  $500\text{ cm}^{-1}$  which may indicate a mono-substituted aromatic ring. Further, the R2567 has a phenyl group as is indicated by the sharp absorption at about  $270\text{ nm}$  in the UV, as shown in Figure 9. Figures 6-10 reveal similar IR spectra for the various materials which are very closely related chemically. However, some differences can be observed. For example, in the  $2860\text{ cm}^{-1}$  region, a weak but very sharp absorption band occurs for both the R2602 (Figure 6) and R2567 (Figure 9). This absorption is not found in any of the other samples. Also, a medium absorption band at  $500\text{ cm}^{-1}$  has little or no definition in RTV-602 but is easily seen in all the other spectra. The absorption bands at  $2860$  and  $500\text{ cm}^{-1}$  may give an indication as to the relative location and abundance of certain C-H groups in the polymer, but confirmation would take much more investigation. The standard absorption maximas for silicone such as shown here are present in the IR spectra but cannot be well identified here because of the sample thickness ( $1250\text{-}1260\text{ cm}^{-1}$  and  $800\text{-}840\text{ cm}^{-1}$  band for Si-CH<sub>3</sub> vibration;  $1090\text{-}1020\text{ cm}^{-1}$  band for Si-O-Si and Si-O-C vibration).

Figures 11-15, giving the UV-VIS-IR spectra, indicate little difference among the resins with the exception for R2567 which shows the presence of an aromatic group. Other differences occurring in the UV-VIS region are likely to be caused by chromophoric contaminants that give many of the silicone their straw or light yellow color. This can be seen by comparing RTV602/L0 (Figure 14) with either R2602 (Figure 12), or SWS 810 (Figure 13). The result is an intensified absorption in the  $400\text{-}250\text{ nm}$  band. This intensified absorption may be eliminated through distillation and/or decolorization processes.

#### 3.2.4. Molecular Weight Analysis

Molecular weight analyses were conducted on the following candidate resins which appeared to have good potential as a replacement silicone:

1. SWS 810
2. SWS V-10
3. SWS V-81
4. MN R-2602
5. GE RTV-12

87433RK

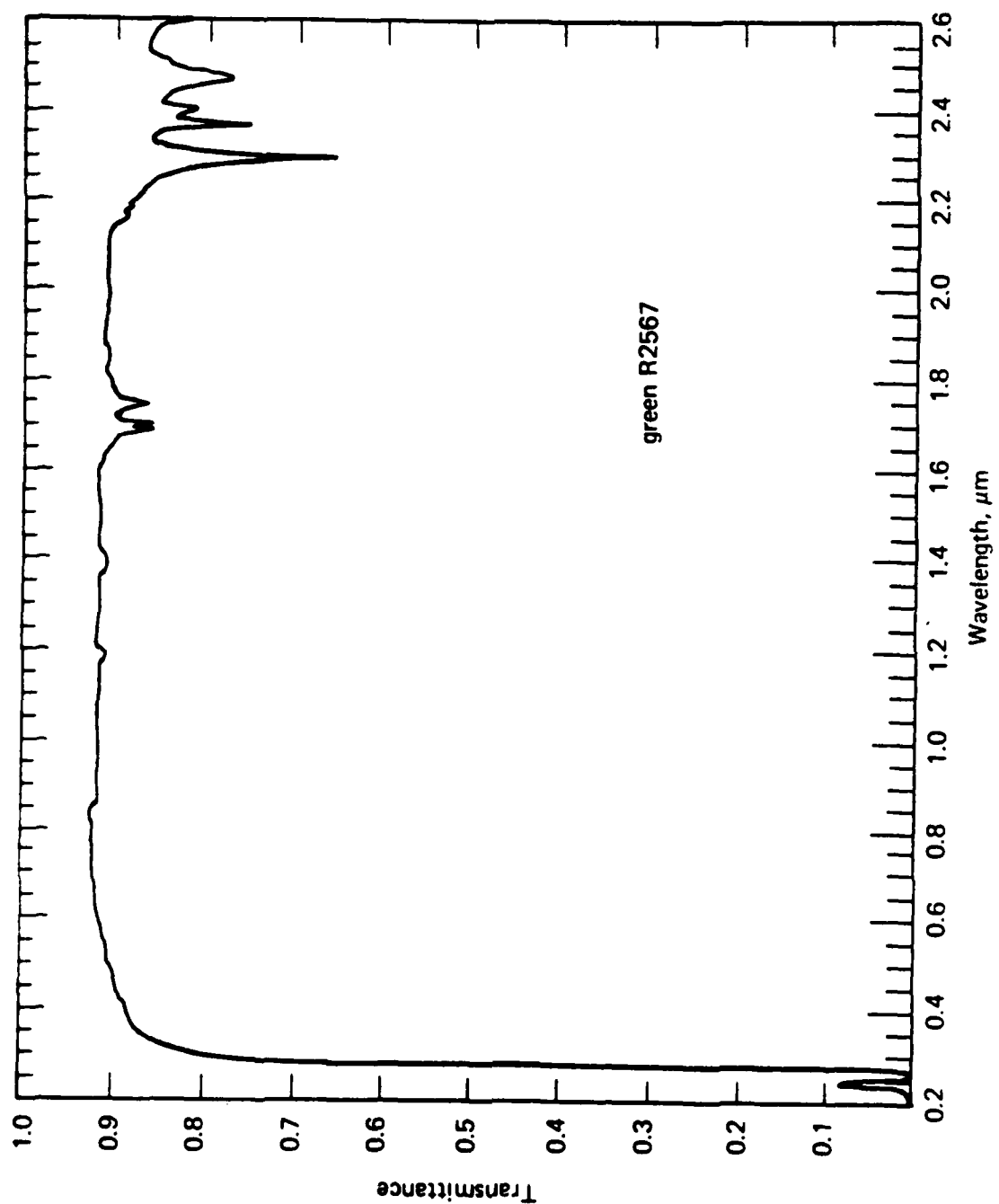


Figure 11. Normal-normal UV, visible and near IR transmittance spectrum of McGhan NuSil R2567 cured resin.

87434RK

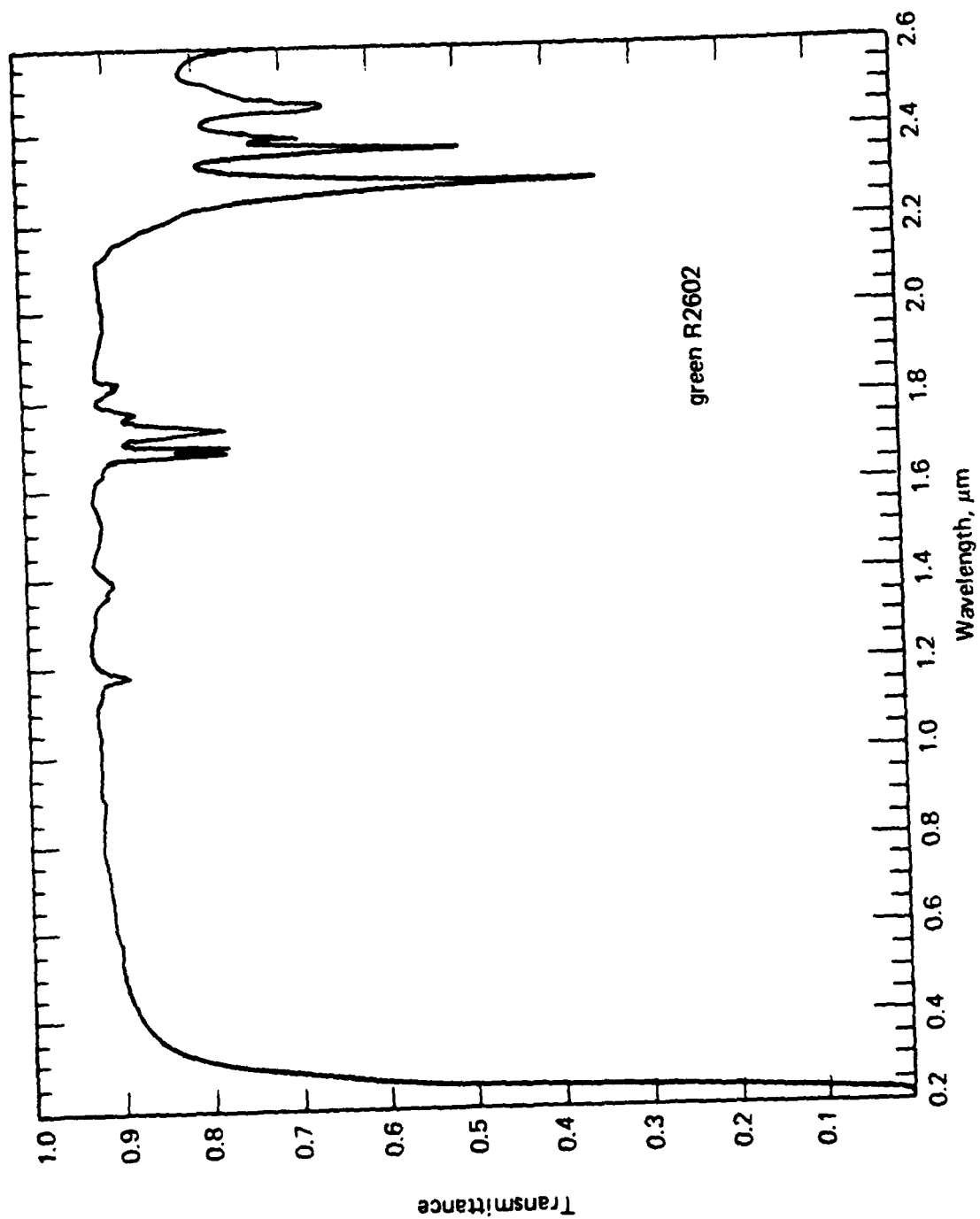


Figure 12. Normal-normal UV, visible and near IR transmittance spectrum of McGhan NuSil R2602 cured resin.

87435RK

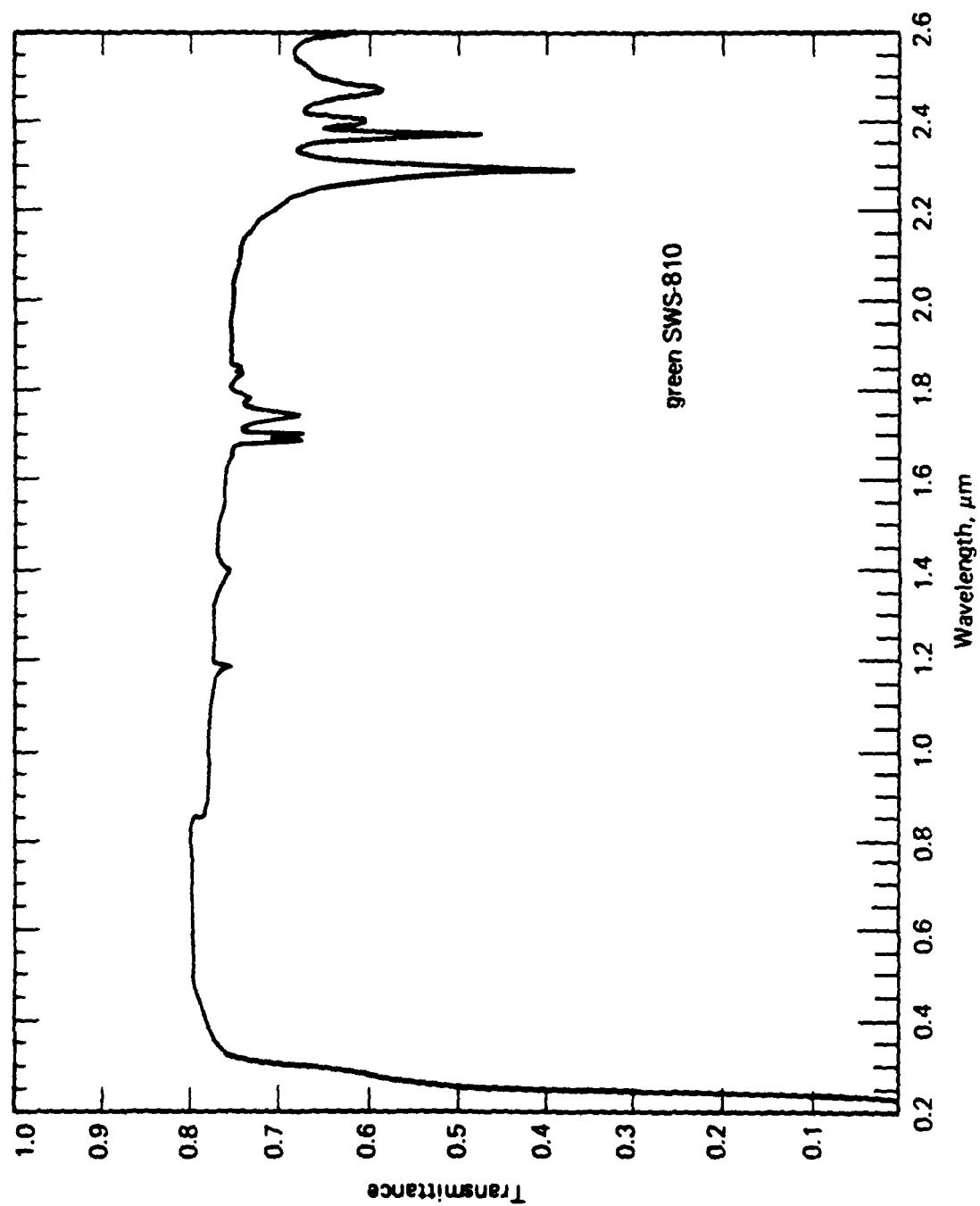


Figure 13. Normal-normal UV, visible and near IR transmittance spectrum of SWS-810 cured resin.

87436RK

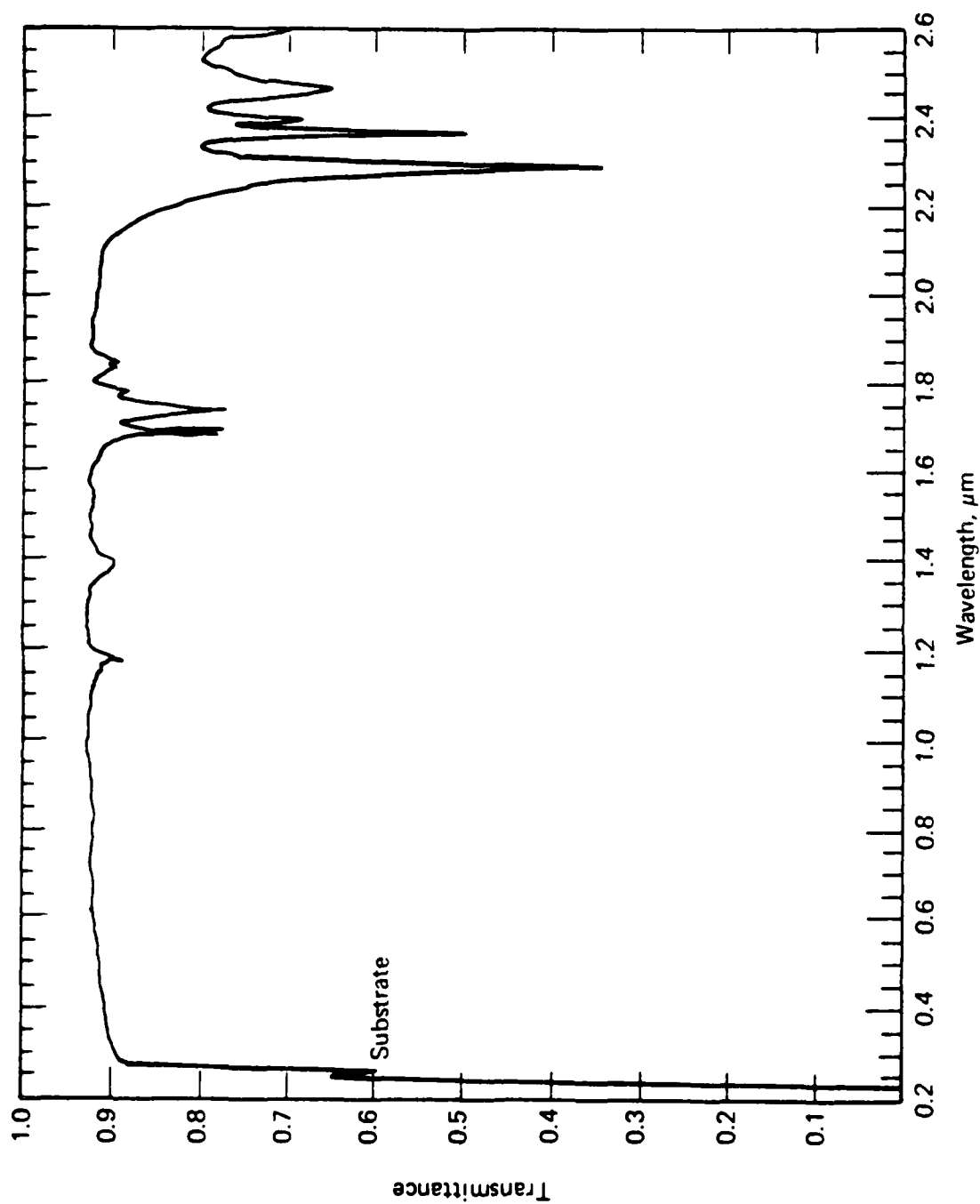


Figure 14. Normal-normal UV, visible and near IR transmittance spectrum of GE RTV-602/LO cured resin.

87437RK

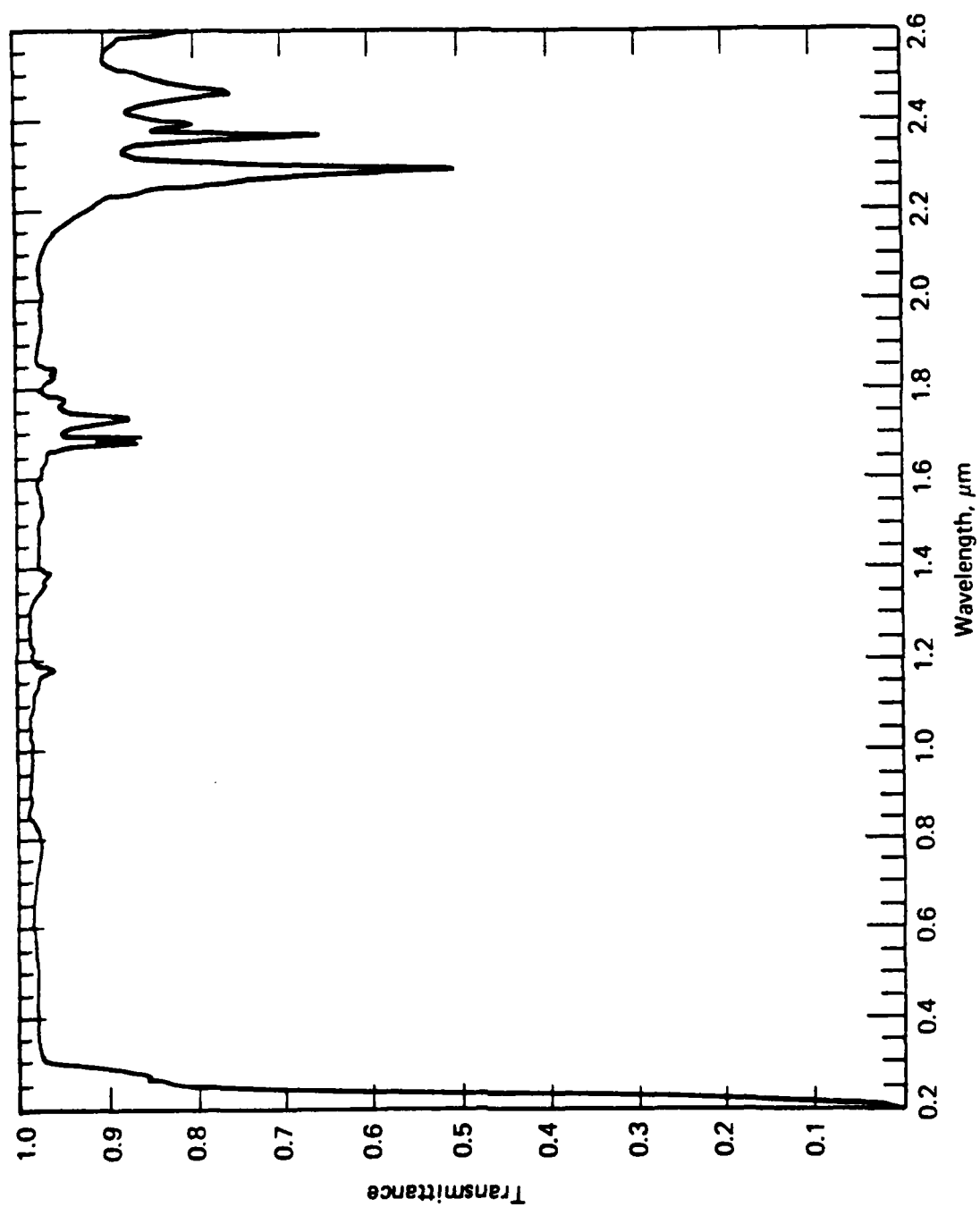


Figure 15. Normal-normal UV, visible and near IR transmittance spectrum of SWS V81 cured resin.

Molecular weight peak maximum and distribution results are presented in Table 5. The purpose of this activity was to: (1) analyze candidate resins for their average molecular weight and molecular weight distribution; (2) determine how similar the candidates are to RTV-602 with respect to their molecular weight and distribution; (3) determine how effective the distillation process is, i.e., the disappearance of lower molecular weight chromatographic peaks; and (4) ensure that significant polymerization of the resin does not occur during the vacuum molecular distillation process.

The molecular weight testing was conducted by Quality Testing Associates (Madison, WI). Gel permeating chromatography (GPC) was used in the testing of the silicone polymers. Toluene was used to reduce the concentration of resin because it is a good silicone solvent and its refractive index is such that it does not interfere with the detection of the silicone.

A series of four (4) Ultrastyrigel columns ( $10^6$  A,  $10^5$  A,  $10^4$  A, and 500 A) were used for this separation. The mobile phase was toluene with less than 0.008% water. The temperature was 90°C and the flow rate was 1.0 ml/min. The samples were made to a concentration of 0.015 g/ml in toluene and were filtered through a 0.45 micron sintered metal filter prior to injection. Each sample was injected in triplicate at a volume of 200  $\mu$ l. The columns were calibrated with a series of narrow dispersity anionically polymerized polystyrene standards.

The materials generally developed a slight increase in average molecular weight after vacuum molecular distillation (Table 5). The results also revealed that the distillation process has been useful in minimizing the amount of low molecular weight material that is present in all of the resins. These values are only representative of the silicone material and will vary from sample to sample as a function of storage, time and environment, and manufacturing process conditions. However, even taking these variables into consideration, the molecular weight distribution of the candidate resins are similar to RTV-602 and therefore would appear to be good substitute candidates.

TABLE 5. MOLECULAR WEIGHT DISTRIBUTION OF SILICONE RESINS

Resin	Predistilled			Post-distilled		
	Peak Max., mol wt.	Mol Wt. High limit	Mol Wt. Low limit	Peak Max., mol wt	Mol Wt., High limit	Mol Wt. Low limit
GE RTV-12	30,368	957,106	203	30,859	1,397,046	899
MN R-2602	28,885	1,709,576	137	33,972	1,925,602	864
SWS V-10	37,992	1,709,576	1,264	43,156	1,280,768	992
SWS 810	37,992	2,030,554	101	40,494	2,479,121	942
SWS V-81	57,403	7,072,198	247	50,580	-	1,264
RTV-602	20,965	541,819	167	21,072	1,315,311	1,342

### 3.2.5. Space Simulation Studies

#### 3.2.5.1. Test Procedure/IRIF II

The IRIF, an acronym for "In-Situ Reflectance/Irradiation Facility," is a multiple-sample ultraviolet-simulation facility processing in-situ hemispherical spectral-reflectance-measurement capabilities shown in Figure 16. The IRIF incorporates an integrating sphere, patterned after one described by Edwards et al.<sup>13</sup> and modified for the Beckman DK spectrophotometers. The major features of the IRIF include:

1. Operation as a module compatible with the Beckman DK spectrophotometer.
2. Operation of the integrating sphere in vacuum at a pressure  $<10^{-7}$  torr.
3. A sample exchange mechanism that maintains, during irradiation, each of the 12 samples in contact with a temperature-controlled sample table and that permits the transfer under high vacuum, of any one of them to the integrating sphere for measurement and the subsequent return to the sample table for continued irradiation.

Standard operating procedures are that, after pump-down, the samples are heated with warm water in the sample table to remove any gross volatiles. Immediately prior to and during irradiation, cold water is supplied to the sample cooling table. Diffuse spectral reflectance measurements are then taken on each of the test samples in the spectral range from 325-2600 nm under high vacuum. Such measurements are repeated after each prescribed test interval as measured in equivalent sun hours (ESH). The following conditions are employed in all IRIF tests:

1. Irradiation rate: 2 sun
2. Vacuum  $<10^{-7}$  torr
3. Sample temperature:  $72 \pm 4^{\circ}\text{F}$

In some tests, an  $\text{O}_2$  bleaching experiment was performed. Upon completion of the post-irradiation reflectance measurements, the system is backfilled with research grade air and the reflectance measurements are repeated to determine recoverable degradation effects.

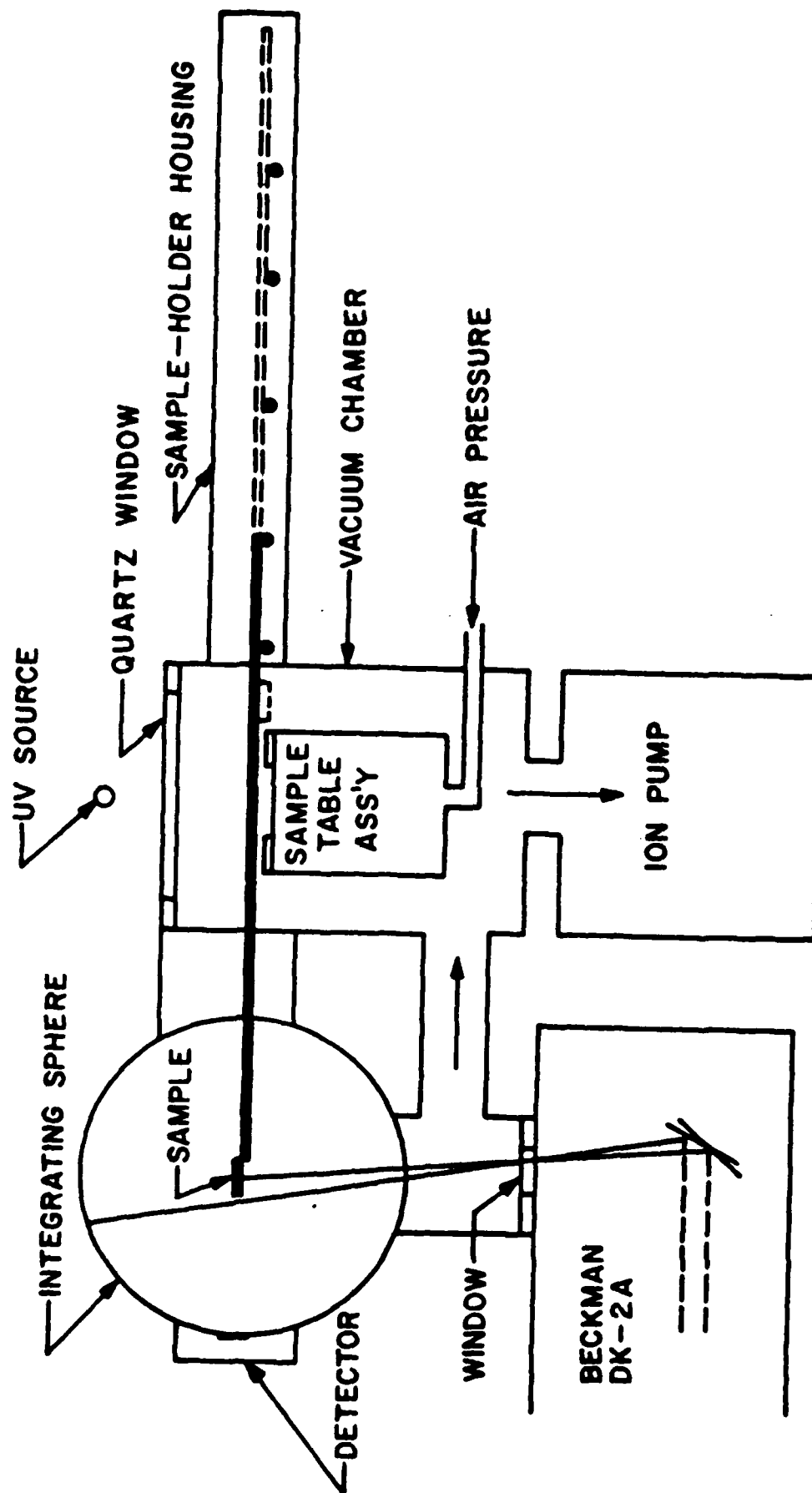


Figure 16. Schematic of UV-irradiation facility with in situ reflectance capability (The IRIF).

### **3.2.5.2 IRIF II/Test Run 1**

A vacuum-UV exposure run containing 2 samples each of the following materials was conducted:

1. RTV-602 control
2. R-2602
3. R-2567
4. SWS-810
5. SWS-V81
6. YB-71 control

The purpose of this run was to examine the response of several selected silicone resins to a space environment. These materials were selected because from initial tests of paint formulations and cure characteristics, they most effectively emulated the behavior of GE RTV-602 silicone resin. The test samples were prepared by deposition of resins onto the surfaces of mirror finished aluminum test coupons. The change in absorptance due to specification and/or coloration of the clear resin reveals degradation behavior.

The primary difficulty with the results of this exposure run was from the excessive degradation of the R-2567 silicone resins as shown in Figure 17. It appears that the R-2567 samples contaminated the other samples, resulting in substantial and inconsistent sample degradation of the other materials.

As can be seen in Figure 17 NM R-2567 underwent severe degradation at  $0.4 \mu\text{m}$  in 50 hr. showing a change in  $\alpha$  of 0.72. RTV-602/L0 for the same duration had a  $\Delta\alpha$  of 0.06. On the basis of these results, the R-2567 was eliminated as a possible binder material.

### **3.2.5.3. IRIF II/Test Run 3**

The purpose of IRIF II-03 space simulation was to determine the effects of UV-vacuum environment on clear silicone resin samples listed in Table 6. The aluminum mirror was used as a reference to detect any foreign material that might become deposited on the samples, and the RTV-602/L0 was used as a standard.

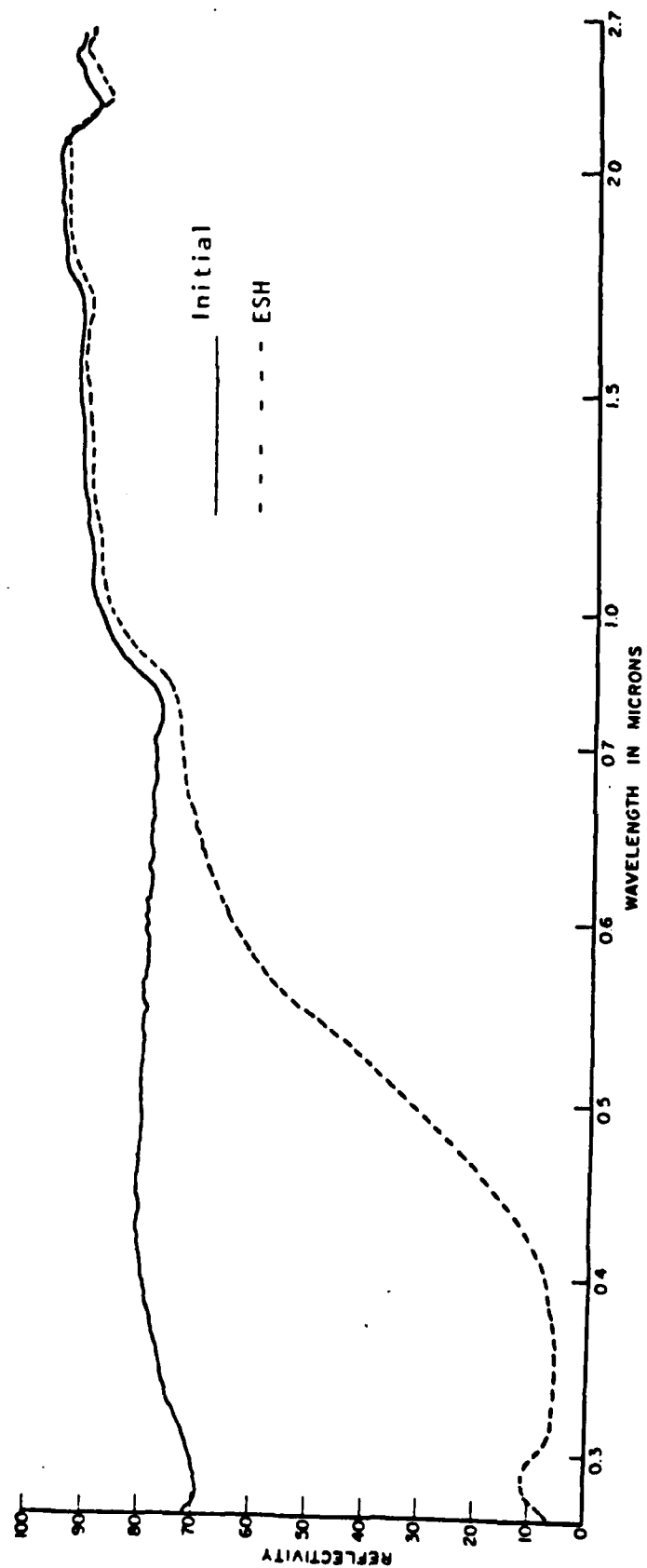


Figure 17. Spectra of MN R-2567 silicone film.

TABLE 6. EFFECTS OF 100 ESH EXPOSURE ON CLEAR SILICONE FILMS

Material	Manufacturer	Solar Absorptance, $a_s$		$\Delta a_s$ , 100 ESH
		Initial	100 ESH	
Al mirror	--	0.12	0.11	.01
RTV-655	GE	0.25	0.41	.16
RTV-615	GE	0.23	0.23	.00
93-500	Dow	0.22	0.22	.00
R-2500	MN	0.17	0.17	.00
V-201	SWS	0.17	0.19	.02
R-2615	MN	0.18	0.18	.00
R-2602	MN	0.18	0.22	.04
V-10	SWS	0.26	0.27	.01
810	SWS	0.25	0.28	.03
V-81	SWS	0.18	0.18	.00
RTV-602	GE	0.17	0.22	.05

After 100 equivalent sun hours (ESH) of UV vacuum exposure, a crack in a manipulator bellows occurred, necessitating termination of the run. Table 6 gives the results of this abbreviated test in which most of the samples showed reasonable stability. The most unstable resin was RTV-655 which had a  $\Delta a$  of 0.16 after 100 ESH. RTV-602, 810, R-2602, and V-201 all showed some degradation. In the case of RTV-602, its past performance does indicate that after the initial degradation the material does tend to level off. Longer tests are necessary to determine if damage saturation does occur.

The silicone resins listed below all demonstrated good UV vacuum stability with a 1-5% change in absorptance between the wavelength of 300-450 nanometers.

- |             |           |
|-------------|-----------|
| 1. RTV-6115 | 4. R-2615 |
| 2. 93-500   | 5. V-10   |
| 3. R-2500   | 6. V-81   |

In addition, V-10 and V-81 also displayed good paint making and paint film curing characteristics, strengthening their position as replacement binders for RTV-602.

Cross-contamination enhanced degradation during this run appeared to be minimal. The reference mirror demonstrated no increased absorptance, and samples adjacent to more highly degraded samples showed no pattern of increased absorptance.

#### **3.2.5.4. IRIF II/Test Run 4**

The purpose of IRIF II-04 space simulation was to continue the IRIF II-03 UV-vacuum environment study. One sample of each resin was employed in this test in order to gain data on a larger number of materials for comparative purposes. When the samples were prepared, care was taken to produce samples with similar thickness ( $\pm 1$  mil) and with minimal scattering from trapped air in the polymer matrix. Sample cleanliness was also maintained to a high degree during production through to sample introduction into the vacuum chamber.

Table 7 gives the results of IRIF II/run 4. All of the resins maintained good UV stability, undergoing some degree of UV vacuum degradation as is illustrated in the reflectance spectra (Figures 18-28). Of the resins tested during this run, the addition polymers listed below demonstrated greater stability than the condensation polymers.

1. R-2615
2. 93-500
3. R2500

The limitations in the use of addition silicone resins is that they tend to be much more difficult to formulate into coatings because they are cure inhibited by many substances, such as a pigment addition. Also, they may require an elevated temperature cure. The addition polymers RTV-615 and V-201

TABLE 7. EFFECTS OF VACUUM UV EXPOSURE ON CLEAR SILICONE FILMS

Material	Manu- facturer	Solar Absorptance, %						After Oxygen Bleach	Change
		0 ESH	100 ESH	200 ESH	300 ESH	400 ESH	500 ESH		
Al mirror	STD	0.11	0.12	0.10	0.12	0.12	0.12	0.01	0.01
RTV-615	GE	0.17	0.20	0.19	0.20	0.21	0.21	0.04	0.01
93-500	DC	0.18	0.19	0.18	0.18	0.18	0.19	0.01	0.01
R-2500	MN	0.17	0.18	0.18	0.18	0.18	0.19	0.02	0.01
V-201	Visloax	0.16	0.18	0.18	0.19	0.19	0.20	0.04	0.02
R-2615	MN	0.17	0.18	0.18	0.18	0.19	0.19	0.02	0.01
RTV-12	GE	0.16	0.20	0.19	0.20	0.21	0.20	0.04	0.00
R2602	MN	0.19	0.21	0.21	0.22	0.22	0.22	0.03	0.00
V-10	SMS	0.25	0.29	0.28	0.30	0.31	0.31	0.06	0.02
810	SMS	0.23	0.24	0.25	0.25	0.26	0.27	0.04	0.02
V-81	SMS	0.22	0.23	0.25	0.25	0.25	0.26	0.04	0.00
RTV-602	STD GE	0.15	0.18	0.19	0.19	0.19	0.20	0.05	0.01

87453RK

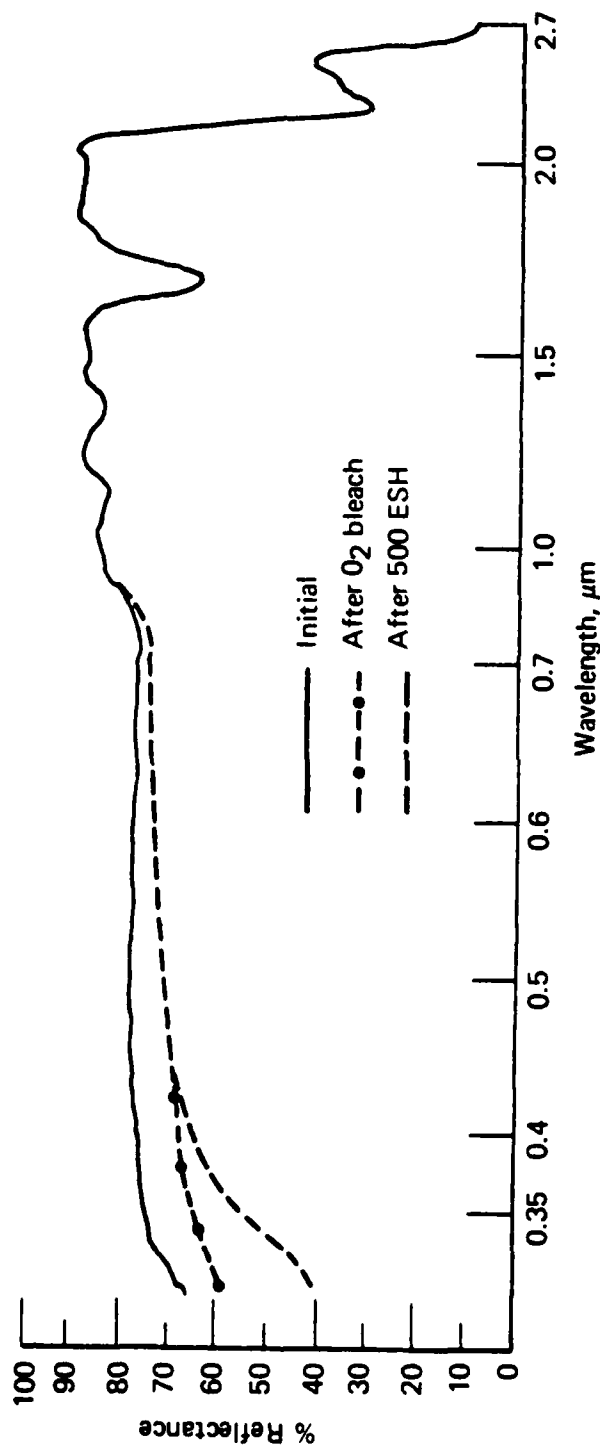


Figure 18. Spectra of SWS 810 silicone film.

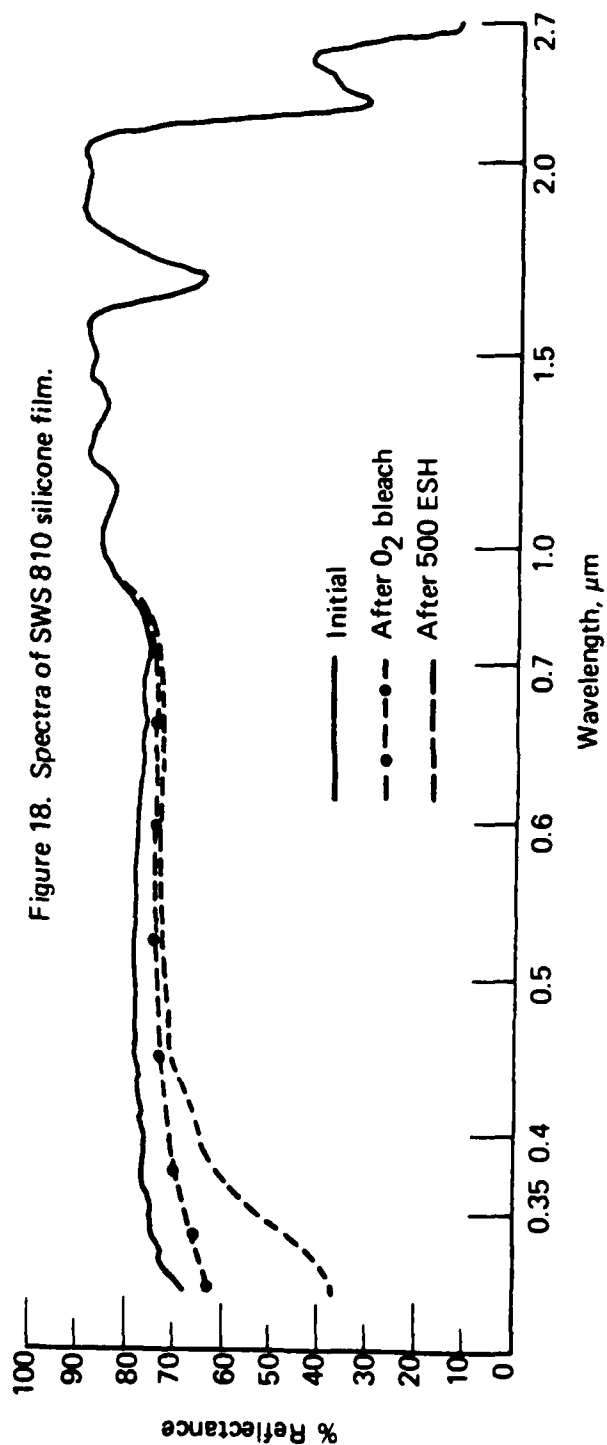


Figure 19. Spectra of SWS V-81 silicone film.

87452RK

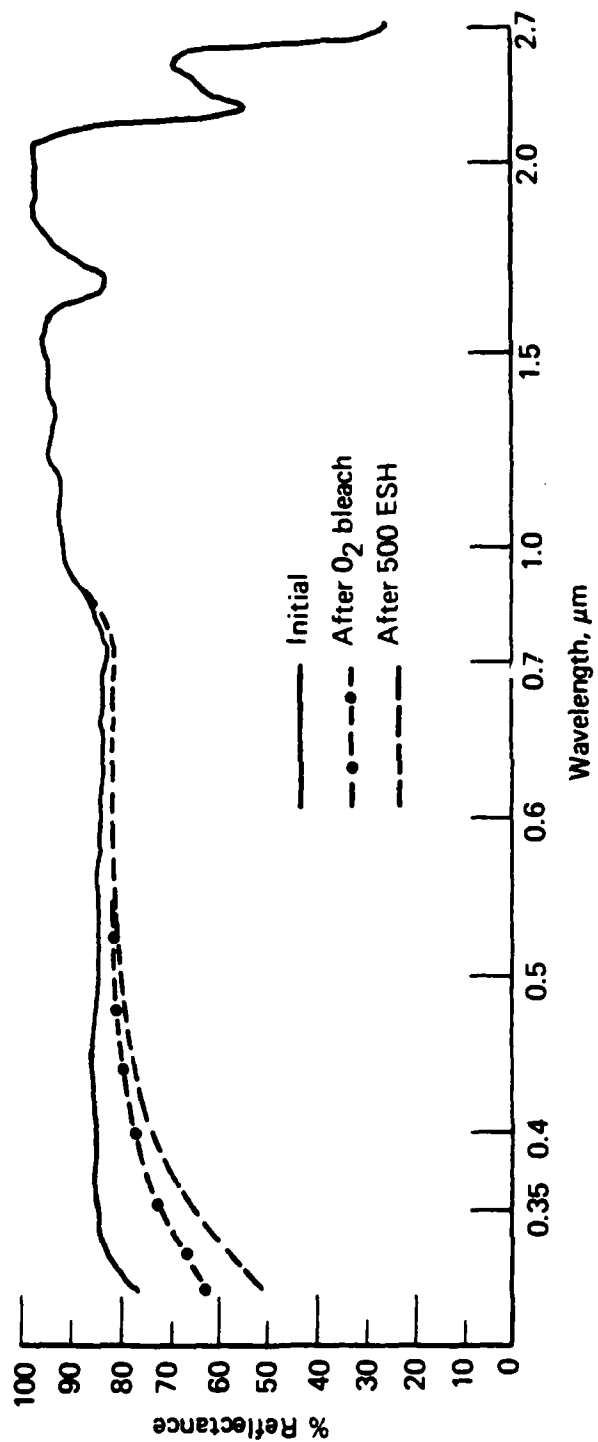


Figure 20. Spectra of Visolox V-201 silicone film.

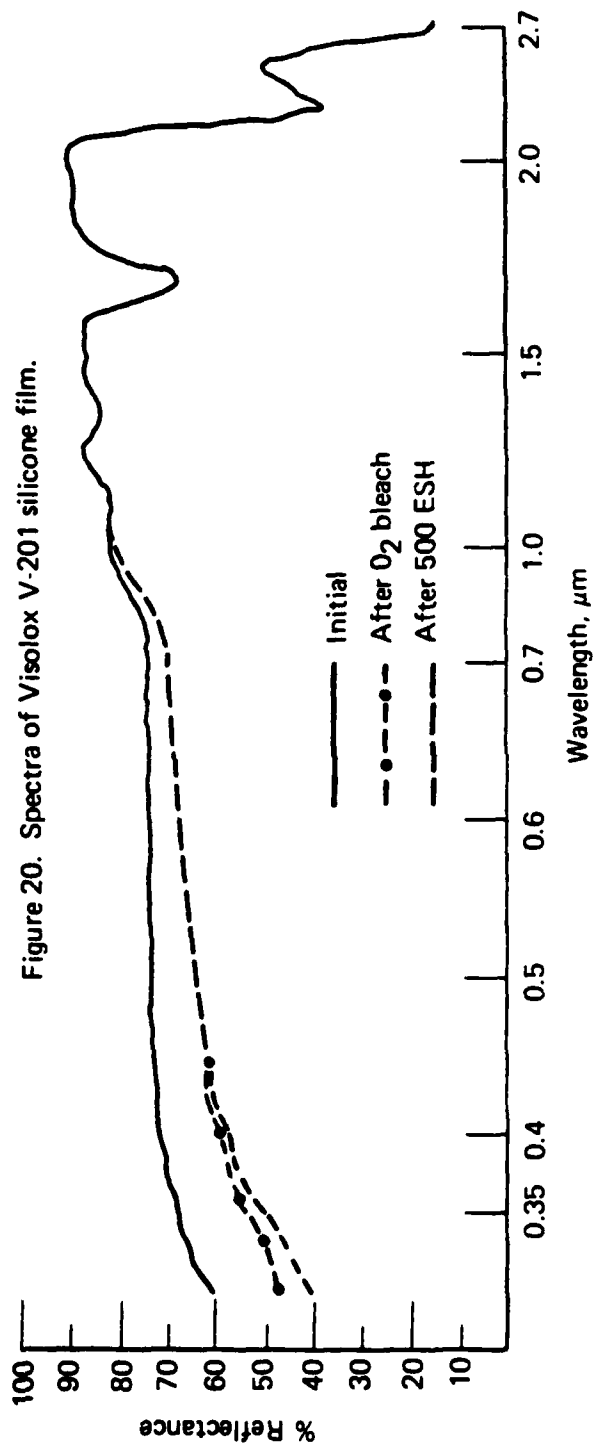


Figure 21. Spectra of SWS V-10 silicone film.

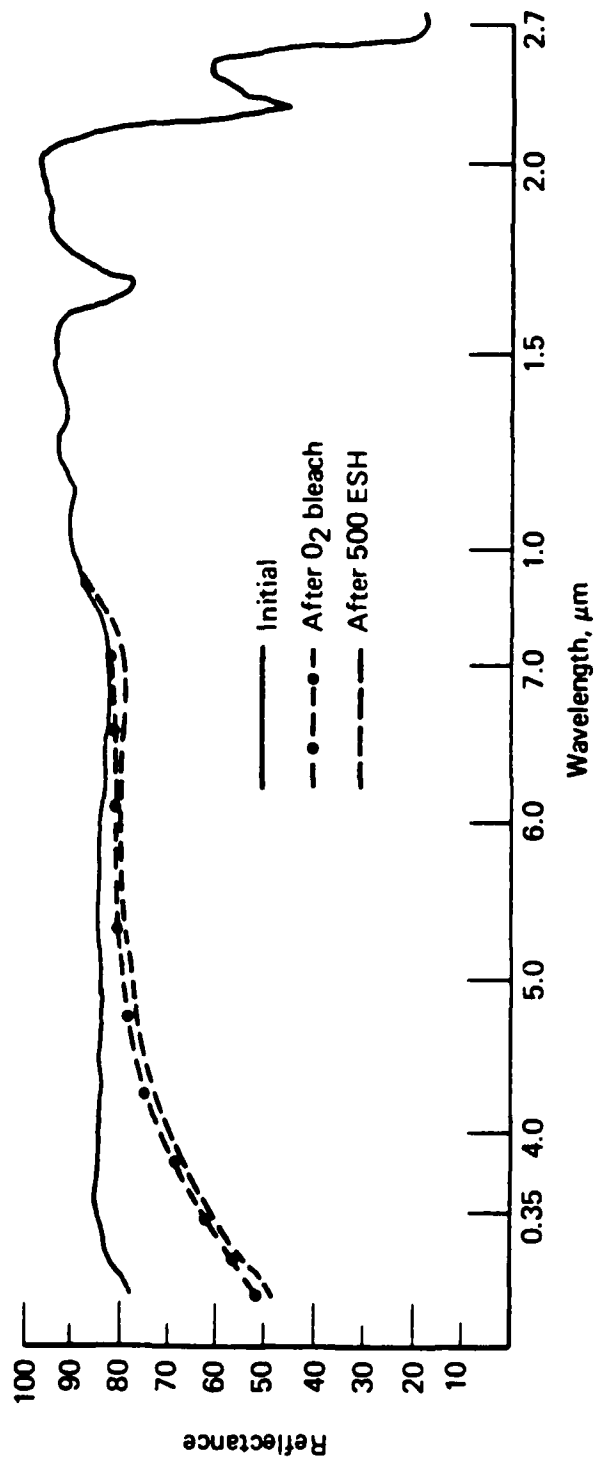


Figure 22. Spectra of GE RTV-12 silicone film.

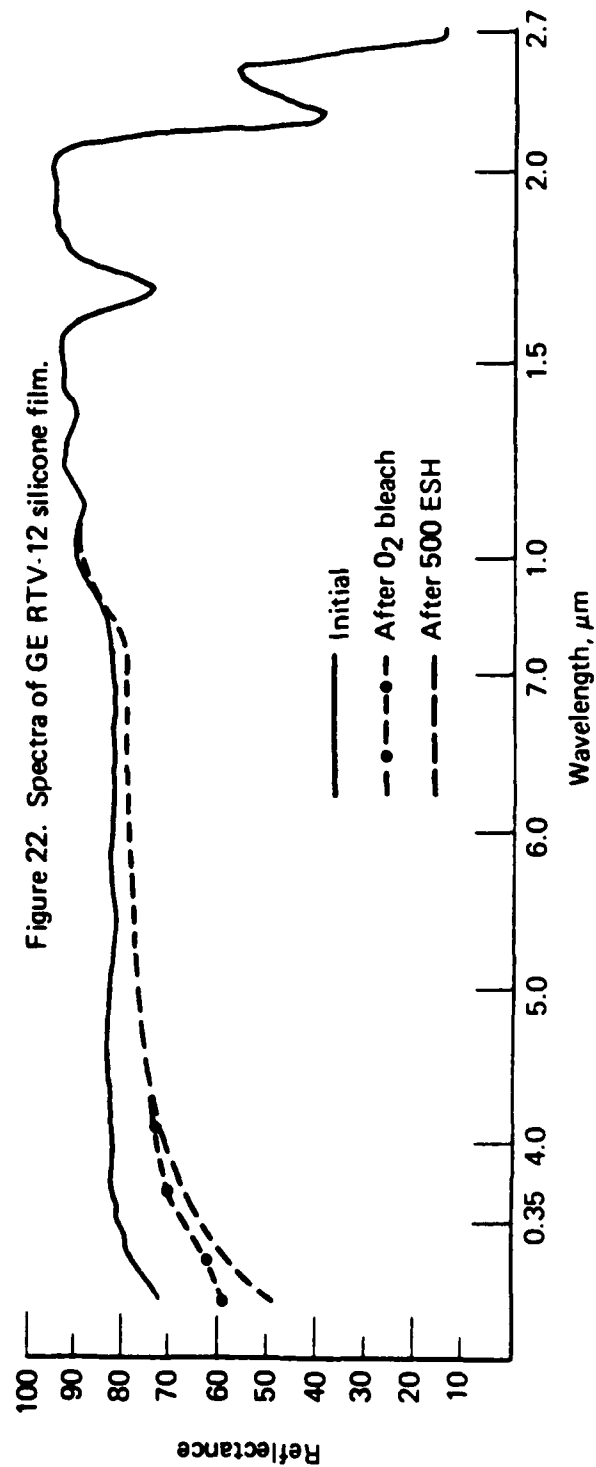


Figure 23. Spectra of GE RTV-615 silicone film.

87439RK

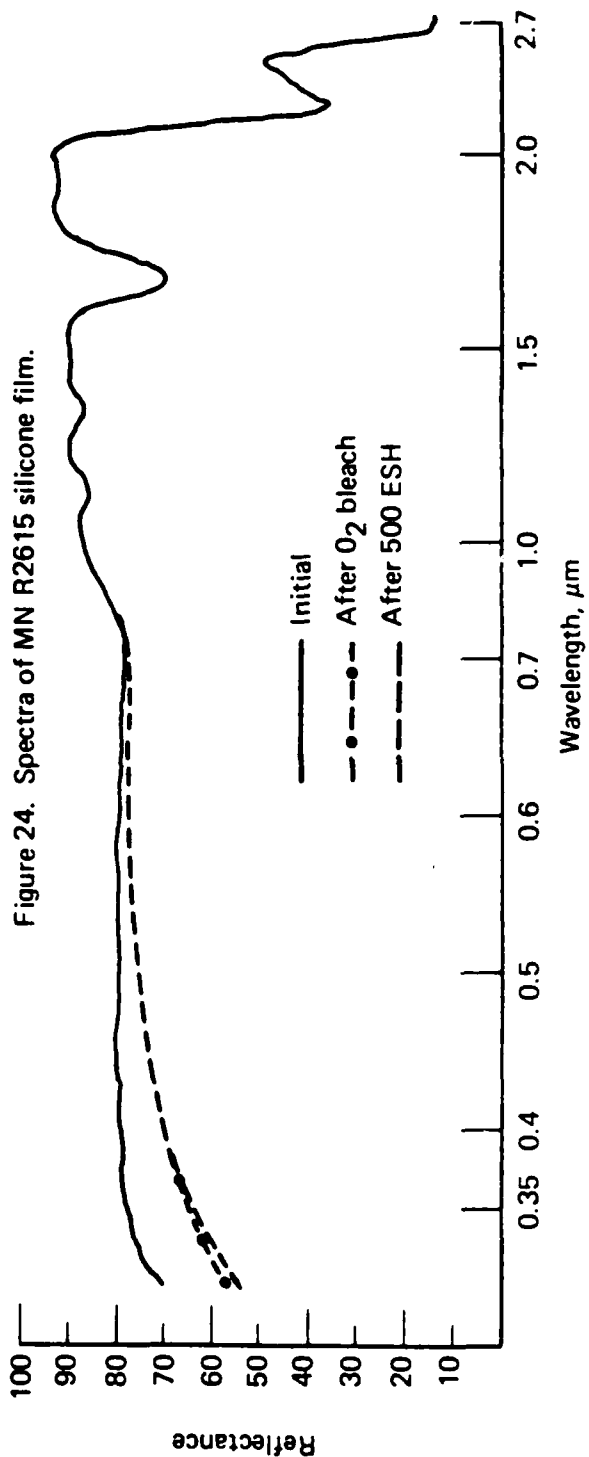
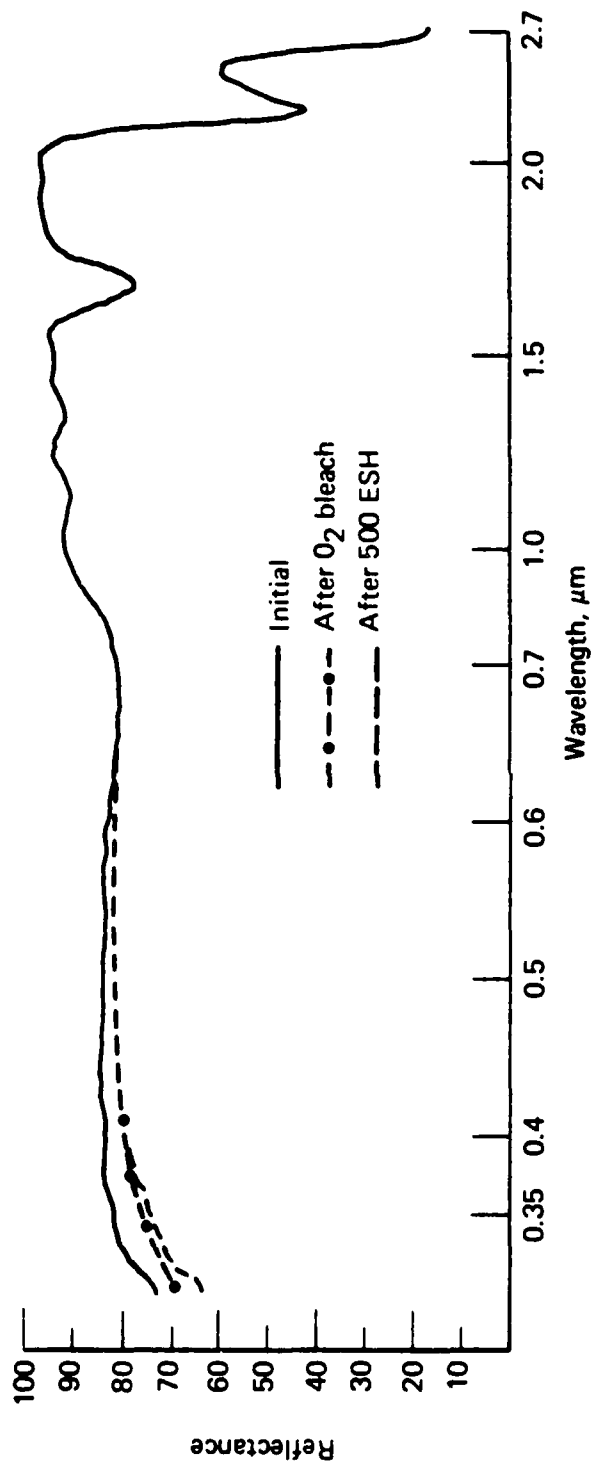


Figure 24. Spectra of MN R2615 silicone film.

Figure 25. Spectra of MN R202 silicone film.

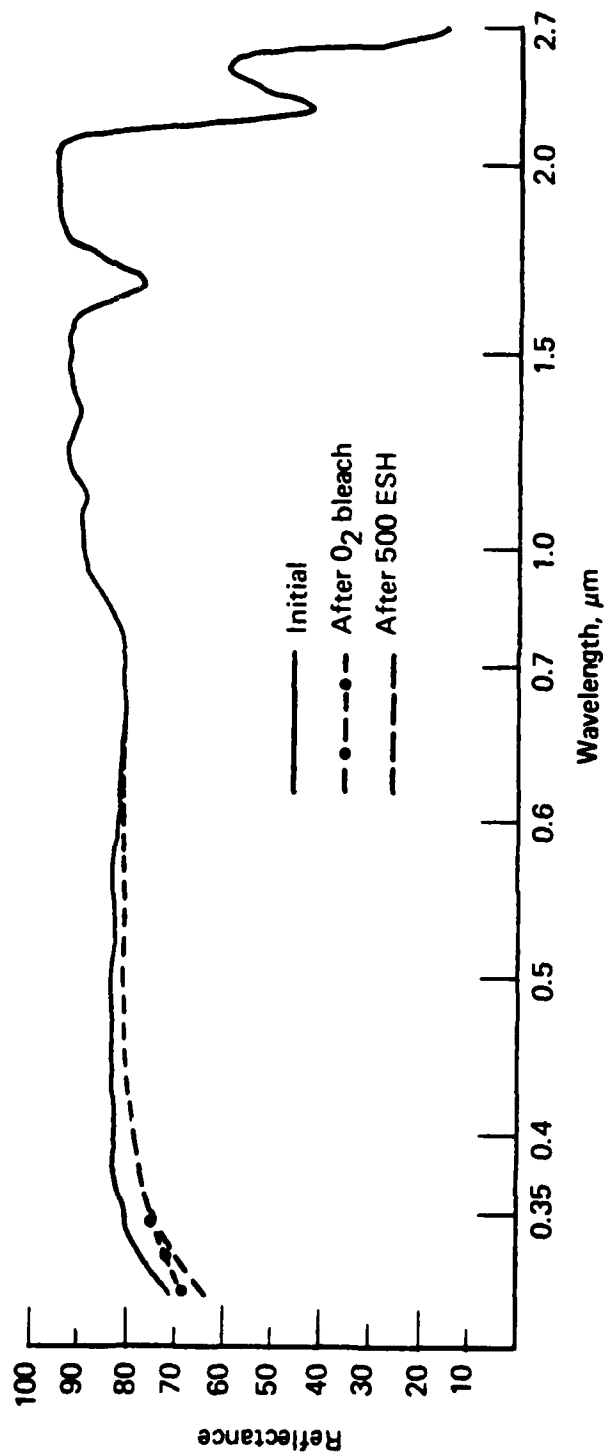


Figure 26. Spectra of DC 93-500 silicone film.

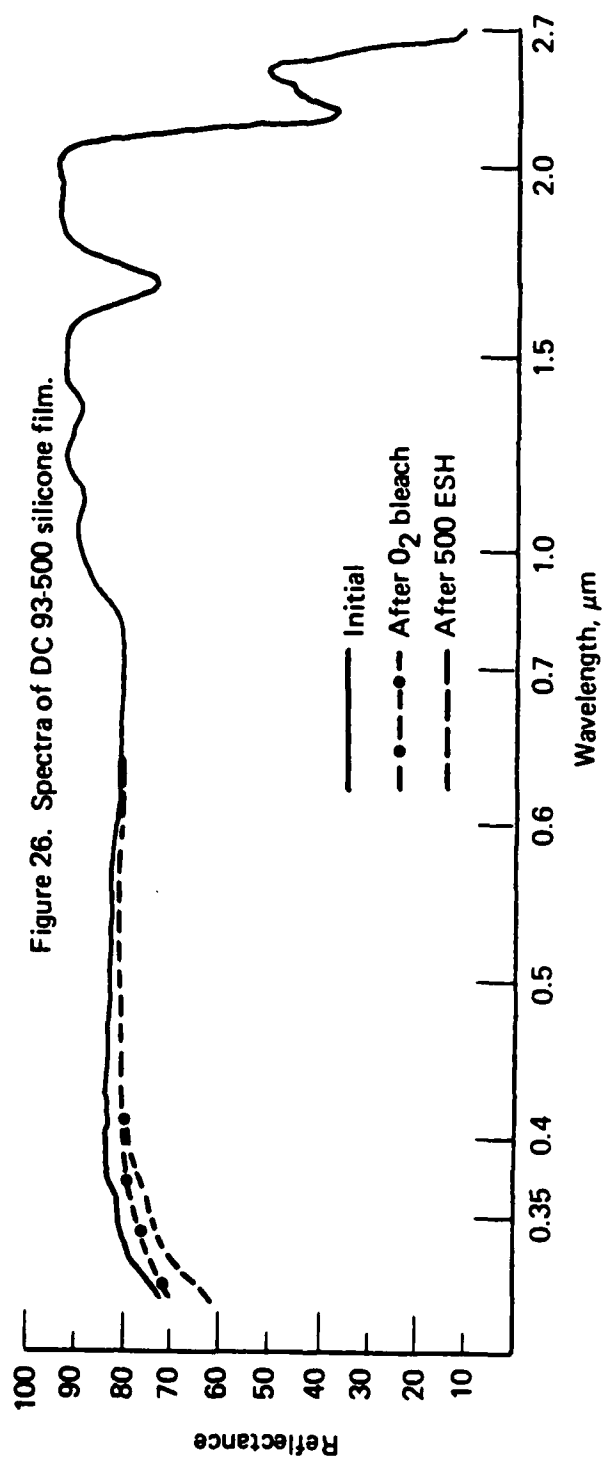


Figure 27. Spectra of MN 2500 silicone film.

87451RK

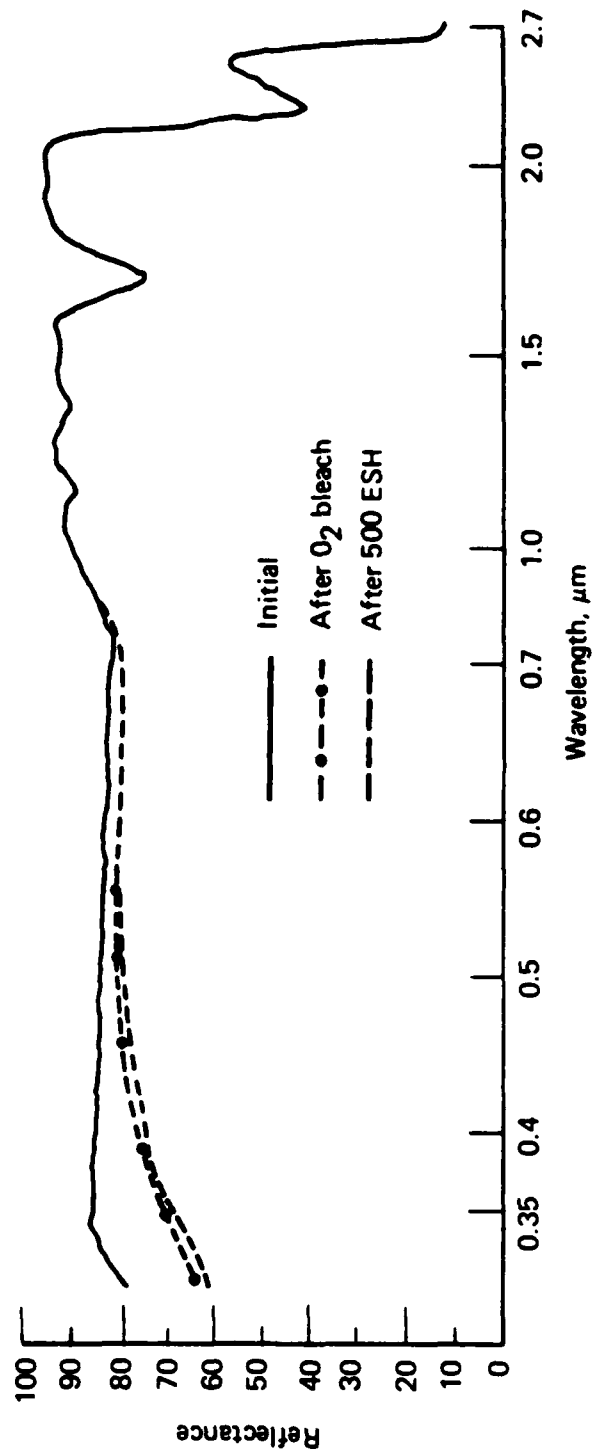


Figure 28. Spectra of GE RTV-602 silicone film.

demonstrated degradation trends similar to the condensation polymers listed here.

- |            |         |
|------------|---------|
| 1. RTV-12  | 4. V-81 |
| 2. RTV-602 | 5. V-10 |
| 3. R-2602  | 6. 810  |

The lesser degree of degradation shown by the resins R-2615, 93-500, and R2500 may be wholly or in part attributed to their greater purity. Most of the other materials show some color and/or turbidity, even when vacuum distilled. In addition, the sample coupons with V-10, V-81, and 810 have a substantial amount of scattering centers in them. It is believed that the scattering centers were produced at the resin/aluminum interface from a reaction between the polymer and the metal surface. As a result of these scattering sites formation, multiple internal reflection of the UV light may have increased their rate of degradation since in run 3 after 100 ESH samples V-10 and V-81 both had a  $\Delta\alpha_s$  of 0.000. The retains show little or no formation of scattering sites. The formation of the scattering sites may be the result of atmospheric water migrating through the resin and acting in conjunction with residual catalyst to produce corrosion at the silicone aluminum interface. At this time, the formation of the scattering centers do not pose a problem because the resins will be incorporated into paints which obtain their reflectance from the scattering effect of the pigment.

Cross-contamination enhanced degradation during this run appears minimal. The reference mirror demonstrated little increased absorptance, and samples adjacent to more highly degraded samples showed no pattern of increased absorptance. The increase and then slight decrease in solar absorptance noted in Table 7 for 200-300 ESH for some of the sample is likely to be the result of spectrometer source malfunction that affected the slit operation.

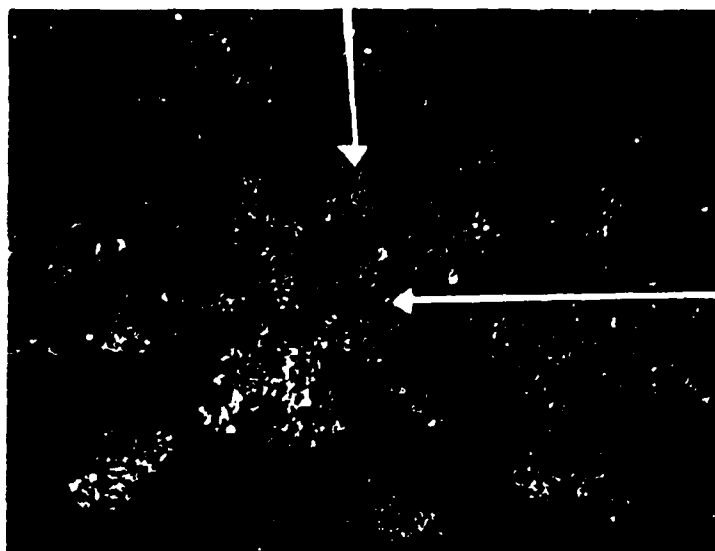
Bleaching studies were also conducted to help in determining the nature of the degradation mechanism, i.e., surface or bulk damage. All of the materials demonstrated some recovery of reflectance as a result of the introduction of air ( $O_2$  bleach) into the system.

The spectra for the silicones all show some degree of recovery (Figures 18 through 28). However, a greater degree of bleaching is evident in samples SWS 810 (Figure 18) and SWS V-81 (Figure 19). This may be related to the formation of numerous vesicles or bubbles that were post-cure formed, possibly as a result of excess catalyst reacting with the polished metal surface.

Figure 29 is an optical photomicrograph of the IRIF coupon with SWS V-81 deposited on the surface. It illustrates (arrow A) a corrosion center and a vesicle cluster (arrow B), both of which are found in great numbers on IRIF samples of V-81, 810, and to a lesser degree on V-10. The vesicles or bubbles also seem to be the centers of coloration when viewed with the aid of a microscope. Further indications show that the vesicles may be in part or wholly responsible for the greater degradation observed for these materials.

Although the phenomenon of corrosion and vesicle formation is apparent and of possible interest for use of the base resins as a potting material or encapsulant, it does not seem to occur when formulated into a coating. The buffer used in the coating formulations ( $\text{ZnSiO}_4$ ) is evidently effective in neutralizing any such effects.

B: Vesicle Cluster

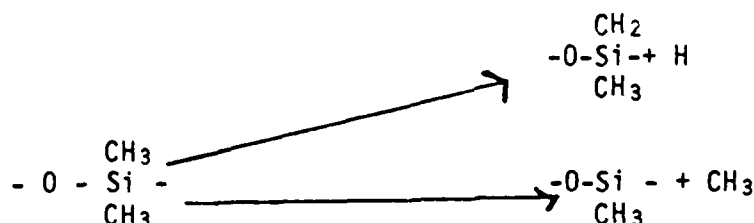


A: Corrosion  
Center

85X

Figure 29. Photomicrograph of SWS V-81 bubble formation due to reaction with aluminum substrate.

The balance of the material evaluated in this run demonstrated only a minor recovery effect, indicating that the damage incurred is not limited to bleachable surface defects caused by charge buildup or atomic exclusion effects, but may be the result of first and second-order bulk polymer degradation, which is nonreversible as shown below:



### 3.3. COATING STUDIES

#### 3.3.1. Formulation Screening Studies

As mentioned earlier in this report, coating studies were conducted concurrently with the resin studies to facilitate cross-feeding of data being generated. The purpose of developing initial coating formulations was twofold; first, it demonstrated which resins were inhibited by the combined components of the formulation, and secondly allowed for the determination of baseline physical and optical properties for the cured coating formulations.

##### 3.3.1.1. Sample Preparation

All of the coatings were formulated using a pigment to binder ratio (PBR) of 2:1 using standard silicate treated zinc oxide pigment, thus simulating S13G/L0. The amount of catalyst per unit mass of resin used in the formulation of the coatings was per manufacturer's published specifications. With McGhan NuSil's R2602 a recommended procedure of adding additional crosslinker was followed for achieving a polymerized coating.

All formulations were ball milled using half and one inch diameter porcelain spheres and cylinders, with a total charge of about 25%. This produced a sufficient grinding and wetting action, which was gentle enough not to cause crystalline distortion of the ZnO lattice. All formulations were milled for 2-3 hr, depending on individual formulation requirements. When the milling was completed, catalyst was added and mixed in thoroughly. The formulation

was then applied to a variety of substrates to a thickness of between 8-10 mils using a Paasche air brush. An air pressure of 30-35 psi was used to apply the coatings. The coatings were then allowed to air cure 24 hr before examination.

### 3.3.1.2. Results

Selected results of the formulation study are presented in Table 8. Item numbers 1 through 8 are experimental formulations utilizing either McGhan-NuSil's R2602 or a stripped version designated R2602/S by IITRI. The best results with R2602 were achieved by using large amounts of crosslinker and catalyst as recommended by the manufacturer. Experiments were also conducted using the R2602/S with the catalysts tetramethylguanidine (TMG) and dibutyltindilaurate (DBTL), each in a 10% solution in toluene (Item Nos. 6 and 7). The main difficulty with R2602 appeared to be a chemical sensitivity to very small amounts of water which may be present in the solvents and/or the pigment, resulting in partial or complete cure inhibition.

Item Nos. 9 and 10, using McGhan NuSil's R2567 (a controlled volatility variant of the R2602) demonstrate much less sensitivity to the coating components as the resin to catalyst ratio indicates. With a resin to catalyst ratio of 100:0.8, the R2567 had one of the lowest amounts of catalyst used in an experimental formulation. It also has produced a cured film which exhibited good strength. However, its strong sensitivity to ultraviolet-vacuum degradation (Figure 17) removed this resin from any extended studies.

Items 11, 12, and 13 were formulated using a one-part resin system incorporating the resin, catalyst (crosslinker), and some solvents into a single package. The one-part systems use either an acetoxo or alkoxy type of reaction. Both systems require atmospheric moisture for the curing mechanism to occur. In the acetoxo system, acetic acid is liberated by the reaction between the resin, crosslinker, and moisture. Alcohol is liberated in much the same manner in the alkoxy system. This type of system is somewhat reactive with atmospheric moisture during the deposition process, and a significant loss of solvent occurs during spray application. Therefore, some percent of the coating is curing before or as it arrives at the substrate, resulting in grainy coatings that have poor to moderate strength.

TABLE 8. SILICONE COATING FORMULATION PARAMETERS

Item No.	IITRI I.D.	Mfr.	Resin Material	Catalyst <sup>a</sup>	Absorbance, <sup>b</sup> $\alpha_{50}$	Relative Strength <sup>c</sup>	Percent Mass of Uncured Coating			Ratio, resin: catalyst
							Catalyst	Solvent	Pigment	Resin
1	I-078A	MN	R2602	Mfr.	0.18	G	1.2	44.8	36.0	18.0
2	I-078R	MN	R2602	TMG	0.18	P	0.4	47.0	35.1	17.5
3	I-080A	MN	R2602/LO	Mfr.	0.19	M	1.2	44.8	36.0	18.0
4	I-080R	MN	R2602/LO	TMG	0.20	P	0.4	47.0	35.1	17.5
5	I-089	MN	R2602/LO	DBTL	--	VP	0.2	39.9	39.9	20.0
6	I-092	MN	R2602/LO	DBTL/ Toluene	0.18	M	0.2	41.0	39.2	19.6
7	I-093	MN	R2602/LO	TMG/T25	0.18	M	0.2	41.0	39.2	19.6
8	I-094	MN	R2602/LO	Mfr.	0.18	M-G	0.4	45.3	36.2	18.1
9	I-079A	MN	R2567	Mfr.	0.17	G	0.2	45.3	36.4	18.1
10	I-079B	MN	R2567	TMG	0.17	P	0.2	46.1	35.8	17.9
11	I-084	DC	I-2577	POR	0.20	M	N/A	43.7	37.5	18.8
12	I-085	DC	3140	POR	0.16	P	N/A	51.6	32.3	16.1
13	I-086	MN	R1009	POR	0.17	P	N/A	72.3	18.5	9.2
14	I-083A	MN	R2615	Mfr.	0.17	VP	1.8	45.5	36.4	18.2
15	I-083B	MN	R2615	TMG	0.17	VP	0.2	46.4	35.6	17.8
16	I-091	GE	RTV-655	Mfr.	0.17	VP	1.4	56.3	28.2	14.1
17	I-095A	SWS	810	Mfr.	0.17	E	0.2	46.3	35.7	17.8
18	I-095B	SWS	810	TMG	0.17	E	0.3	46.8	35.3	17.6
19	I-096A	SWS	V-81	Mfr.	0.19	G	0.3	46.8	35.3	17.6
20	I-096B	SWS	V-81	TMG	0.19	G	0.3	46.8	35.3	17.6

<sup>a</sup>Mfr.= manufacturer's catalyst, TMG= tetramethylguanidine, DBTL= dibutyl tin dilaurate, POR= part of resin.<sup>b</sup>Solar absorbance for air mass zero.<sup>c</sup>VP= very poor, P= poor, M= moderate, G= good, E= excellent.

Items numbered 14, 15 and 16 are all addition cure two-part silicone systems. An addition cure silicone system consists of base resin (part A) and a specific curing compound (part B), generally mixed in a 10:1 ratio. The advantage of the addition polymer is that it does not produce any curing byproducts like water, that condensation cure polymers may produce. They are highly susceptible to cure inhibition from chlorinated or sulfonated compounds as well as amines, organometallics, and water. The attempts to produce a viable coating were unsuccessful, even though it has been suggested by GE that RTV-615 could be a replacement for RTV-602. The water that may be present in the solvents or in the S13G pigment may preclude any possibility of the system curing unless special precautions are taken to remove the absorbed water from the components. In addition, the basicity of the pigment or the metallic portion of the pigment molecule may in itself cause cure inhibition.

Among the various early formulation, Item Nos. 18-21 were the best coatings. These resins are all dimethylsiloxanes and have physical, optical, and chemical properties very similar to RTV-602. Furthermore, the SWS-810 resin material seemed to be tougher and to have greater tear resistance than the RTV-602 material. The amount of catalyst required to produce a coating containing this resin is very small, as is shown by the resin to catalyst ratio of 100:03 in Table 7. The data for SWS-810 and SWS-V81 coatings were initial results, and this ratio was reduced in later formulations. In addition, the cure time required to obtain a tack free coating surface was less than 20 minutes. A duration of approximately one hour is usually required for a coating using RTV-602 as the binder. The SWS-810, V81 formulations may be more stable than the RTV-602/L0 because of the potentially lower amounts of catalyst required to produce a tough film. This would tend to minimize the potential for volatilization of catalyst, which could undergo photodegradation and redeposition onto the surface, degrading the optical properties over a wider area where secondary surface reactions may occur. Although the data for SWS V-10 resin is not shown in this table, it appeared to be very similar to SWS-810, with somewhat better optical properties.

More advanced formulations using the most promising resin materials are shown in Table 9. These resins were subjected to distillation (See Section 3.2.1) to minimize the amount of included volatile components. The coating formulation produced from RTV-12A/S (Items 1 and 4) had generally good

TABLE 9. SILICONE COATING FORMULATION PARAMETERS

Item No.	IITRI I.D.	Resin		Catalyst <sup>a</sup>	Relative Strength <sup>c</sup>	Percent Mass of Uncured Coating		Ratio, resin: catalyst
		Mfr.	Material			Catalyst	Pigment	
1	J-068	GE	RTV-12A/L0	TMG	M	.015	49	100:08
2	J-069	MN	R2500	Mfr.	VP	2.0	39	10:1
3	J-071	DC	184/L0	Mfr.	VP	2.0	47	10:1
4	J-073	GE	RTV-12A/L0	TMG	E	.015	45	100:08
5	J-079	SWS	810/L0	TMG	P	.3	45	100:1.25
6	J-080	SWS	V-81/L0	TMG	VP	.3	45	100:1.25

NOTE: All resins formulated with K-99 reducer except Item No. 4 which was formulated with toluene.

characteristics. Item 4 is the same as 1 with the exception of using toluene as the thinner. It produced an excellent coating with improved sprayability and strength. In addition, it required only a small amount of catalyst to effect a good cure of the material.

MN R-2500 and DC 184/S (Items 2 and 3, respectively) both seemed to have similar problems in curing: (1) cure inhibition by the thinner system, and (2) possible insufficient catalyst as supplied by manufacturer to effect 8-24 hr cure. The use of additional platinum catalyst was recommended by the manufacturers. It was indicated that the addition of 1-2 drops per 100 grams of base should produce a well-cured film at room temperature, even in the presence of fillers and solvent/thinner systems.

Items 5 and 6 incorporating SWS materials 810 and V81 demonstrated poor film characteristics. The material as supplied by the manufacturer contains crosslinker as an integral component of the resin. An appreciable amount of this volatile crosslinker is lost during molecular distillation, and even more is lost during the spray deposition process, resulting in films that will not cure at room temperature. However, this potential problem has been obviated in the SWS resins (including V-10) by obtaining the base resin free of the crosslinker. After the resin has been molecularly distilled, the crosslinker is added to the overall formulation.

#### **3.3.1.3. Summary**

The silicone resins which exhibited behavioral characteristics similar to RTV-602 were: (1) SWS V-10; (2) GE RTV-12; (3) SWS 810; (4) SWS V-81; and (5) MN R-2602. Based on the results of UV vacuum exposure, TWL/VCM, and the other criteria, the SWS silicone materials exhibited the best properties. Furthermore, the V-81 and V-10 materials showed the best results of any of the silicones.

The twenty silicone resins shown earlier in Table 1 were chosen for evaluation since they met the initial requirements of room temperature cure and high visible clarity. The one-part silicone resins listed in Table 10 were eliminated from the program because:

1. The resins tend to be turbid and yellow and have high viscosity.
2. Experimental formulations exhibited poor spraying

TABLE 10. MATERIALS DROPPED OR GIVEN REDUCED PRIORITY

<u>Manufacturer</u>	<u>Material Designation</u>	<u>Dropped</u>	<u>Reduced Priority<sup>a</sup></u>
McGhan Nusil Corp. (Carpinteria, CA)	R-2500		AC
	R-2567	SD	
	R-2615		AC
	R-1009	OP	
General Electric Co. (Waterford, NY)	RTV-615		AC
	RTV-655		AC
	RTV-670		AC
Dow Corning Corp. (Midland, MI)	R-6103		AC
	93-500		AC
	Sylgard 186	V	
	Sylgard 184		AC
	Sylgard 182		AC
	DC-3140	OP	
	3-6527		AC
	DC-2577	OP	

<sup>a</sup>Reasons: AC= additive cure; SD= severe UV vacuum degradation; OP= one part system; and V= high viscosity.

characteristics and clogging of spray equipment occurs frequently, even with additional solvents added.

3. They tended to produce coatings that were powdery and of poor strength.
4. Their potential shelf life is minimal because these silicone compounds are one-part kits with no catalyst required to be added for curing to occur.
5. Because of the aforementioned reason, this class of silicones do not emulate the characteristics of RTV-602 well and would therefore make a poor replacement material.

The addition cure type silicones were given reduced priority because of their tendency toward cure inhibition from pigment material and solvents. The formulations that could be cured tended to do so only at reduced pigment concentrations, using ether as the solvent.

These screening studies indicated that incorporation of dimethylsiloxanes yielded the most promising formulations. It was on this basis that the number of candidate materials was narrowed to the SWS materials, and RTV 12 as substitutes for RTV 602 in S13G/L0.

### **3.3.2. VCM/TWL Studies**

As pointed out in Section 3.2.2., VCM/TWL tests were conducted at both Ball Aerospace and McGhan NuSil. As the earlier discussion showed, Ball's data showed somewhat greater outgassing than did McGhan NuSil's. Experimental batches of thermal control (TC) coatings were produced using the following stripped resins: GE RTV-12, MN R2602, SWS V-10, V-81, and 810. The experimental coatings were made with a pigment-to-binder ratio (PBR) of 2:1 and were tested for VCM/TWL. The resins used in the paint films had been distilled under the conditions outlined earlier in this report in Table 2. Catalyst and solvent were then added, and the mixture was deposited onto a Mylar substrate using air spraying. Spraying conditions were an air pressure of approximately 30-35 psi with an average distance to the substrate of 10-12 in. The paint films were allowed to air dry at room temperature for two weeks. An adequate size sample (approximately 2 x 3 in.) was then removed from the substrate and submitted for testing.

The testing of the paint films was carried out by McGhan NuSil Corporation in accordance with E-595-77 and NASA SP-R-0022A specifications. The test

consists of washing the surface of the samples with Freon to remove any foreign material that might have accumulated during transit. Then, four samples of each material were preconditioned by placing them in a 23°C and 50% relative humidity environment for 24 hr and then weighed. Next, the test samples are exposed to a 125°C,  $10^{-5}$  torr environment for 24 hr. The collector is maintained at a temperature of 25°C for the collection of volatile condensable materials. The condensing element is then weighed, and the amount of material deposited is determined. The sample material is then reconditioned at 50% relative humidity for 24 hr and reweighed to determine total mass loss (TML).

The results of the VCM/TWL tests are presented in Table 11. The data show that all of the TC coatings have a TWL of 0.30% or less with the exception of R-2602 with a TWL of 0.66% which is still well below the 1.0% criterion.

TABLE 11. VCM/TML ANALYSIS OF PIGMENTED SILICONE FILMS

<u>Resin Material</u>	<u>Manufacturer</u>	<u>IITRI ID</u>	<u>% TML</u>	<u>% VCM</u>	<u>Comments</u>
RTV-12(S)	GE	J-142	0.22	0.01	No visible condensate
810(S)	SWS	J-140	0.25	0.00	No visible condensate
V-10(S)	SWS	J-153	0.30	0.00	No visible condensate
V-81(S)	SWS	J-151	0.24	0.01	No visible condensate
R-2602(S)	MN	J-143	0.66	0.00	No visible condensate
RTV-602(S)	GE	Avg	0.45	0.05	

The VCM results for these samples were excellent. All of the samples are well below NASA specifications of 0.1% VCM with a maximum VCM value of 0.05% for V-81 and RTV-12, 0.005 for the remaining materials.

The performance of the coatings with processed resins showed steady improvement during the course of the program. This was the result of very careful molecular distillation in some cases, requiring two or more cycles through the system in order to obtain a final system pressure in most cases or less than one micron. With this significant reduction in VCM/TWL values, it

was evident that a TC coating produced from any of the candidate resins would be acceptable from an outgassing standpoint based on present standards.

### **3.3.3. Physical Properties**

#### **3.3.3.1. Tensile Strength**

Tensile tests were conducted on five experimental paint coatings incorporating the most promising resins, i.e., RTV-12, SWS V-10, SWS V-81, SWS 810, and MN 2602. All resins tested were first stripped of volatile material before being blended with pigment and X-99 thinner. All five coating systems employed a 2.1 to 1 pigment (S13G) to binder ratio. Along with the five candidate coatings, a batch of S13G/L0 was also formulated and was used as a standard to compare with the other paint formulations.

To produce tensile specimens that are relatively flat from side to side and along their lengths, the experimental coatings were cast into large sheets rather than being cast into individual tensile specimen molds. The paint batches were cast on a 6 by 7 in. aluminum plate covered by Tedlar film to a thickness of 0.1 inch. After the experimental paint had cured for two days, tensile specimen stamp was used to obtain ten specimens from each paint film. All tensile specimens were allowed to cure an additional two weeks before testing to assure a complete cure. Specimens were measured for width and thickness, and minimum dimensions were used to determine cross-sectional area. Specimens were placed in the grips taking care to align the long axis of the specimen and the grips. The maximum load carried by the specimen during testing was the load at the moment of rupture.

The testing machine employed in evaluating the experimental paint films was an Instron Universal Testing Instrument Model (TT-D). All tensile tests were conducted using a "C" tensile load cell and a 0-22 lb load scale in accordance with ASTM standard D-638-61-T. The crosshead testing speed selected for the paint films was 20 ipm. The grips employed were self-aligning so that the long axis of the test specimen coincided with the direction of the applied pull through the centerline of the grip assembly.

Results of these tensile tests are presented in Table 12. All values are average values for ten specimens for each coating. Percent elongation at the moment of rupture for each tensile test was measured from the chart readout. Gauge length for all of the tensile specimens was 0.75 in.

The various coatings formulations exhibited strength and elongation behavior similar to that for the RTV-602/L0 standard. All of the candidate systems appear to possess cohesion properties adequate to resist damage from normal handling and performance requirements. The relatively high strain-to-failure behavior suggest good elastomeric qualities for these coatings.

**TABLE 12. TENSILE TEST RESULTS FOR SILICONE PAINT COATINGS**

Resin Material	Manf.	psi	% Elongation
RTV-602/L0	GE	704	75
RTV-12/L0	GE	727	32
R-2602/L0	MN	684	68
V-10/L0	SWS	440	87
V-81/L0	SWS	712	74
810/L0	SWS	481	40

NOTE: Data  $\pm 10\%$

### **3.3.3.2. Abrasion Resistance**

Abrasion tests were performed on five experimental paint formulations using a Taber Abraser. Test samples were produced employing the following resins: RTV 12, SWS V-10, SWS V-81, SWS 810, and MN 2602. All resins were first stripped of volatile material before being blended with pigment and X-99 thinner. All five resin groups to be evaluated employed a 2:1 pigment-to-resin ratio and S13G pigment was used in each of the five coatings. A batch of S13G/L0 paint was also prepared to be used as a standard for comparison purposes.

Abrasion test specimens consisted of coatings on a 4 by 4 in. by 3/16 in. thick aluminum substrate with a 1/4 in. hole in the center to attach the plate to the specimen holder. Before the experimental paints were sprayed onto the plates, each plate was thoroughly cleaned with solvents, primed with SS-4044 silicone primer, and then weighed. Five aluminum plates were painted with each of the six coatings, for a total of thirty abrasion test plates. At

least one week was allowed for the resin to thoroughly cure and the paint thinner to evaporate. The painted abrasion samples were then trimmed of excess paint at the edges and then re-weighed to determine coating weight. The abrasion resistance of the five experimental paint formulations along with the standard S13G/L0 paint, were evaluated using a Taber Model 503 Abraser in accordance with ASTM Standard D-1044-56.

Coatings produced from the following resins (V-10/L0, V-81/L0, 810/L0, and RTV-12/L0) exhibited weight losses smaller than S13G/L0 (Table 13). These results indicate that coatings formulated with any of these resins as binder should be as resistant to handling and cleaning damage as the present S13G/L0 material. Paint samples produced utilizing the binder resin R2602/L0 resulted in the highest material removal of all of the coatings. This coating's abrasion resistance is slightly less than that of the S13G/L0 with a 19% increase in material loss. However, all of the candidates exhibited adequate resistance to abrasive wear.

**TABLE 13. ABRASION TEST RESULTS FOR SILICONE PAINT COATINGS**

Material	Manf.	Avg. Weight Loss, g	% Change in Weight, Relative to S13G/L0*
V-10/L0	SWS	0.2520	-35
RTV-12/L0	GE	0.2092	-46
R2602/L0	MN	0.4762	+19
V-81/L0	SWS	0.2099	-46
810/L0	SWS	0.2144	-45
RTV-602/L0	GE	0.3880	

\*A minus value indicates greater abrasion resistance, and a plus value lower abrasion resistance, as compared to S13G/L0.

### **3.3.3.3. Adhesion**

Five samples of each candidate paint were prepared for adhesion tests on 4 x 4 in. 6061 aluminum substrates which were cleaned and abraded lightly with a 3M Scotch Brite cloth. GE, SS-4044 primer was then applied to the surface of the aluminum coupons to a thickness of less than 0.4 mils, allowed to dry

for 2 hours, and the candidate coatings were applied by spraying to a thickness of 9-11 mils.

The samples were cured for 7-10 days prior to testing. The adhesion test consisted of immersing the sample in liquid nitrogen, and then removing the sample after it had reached equilibrium temperature with the LN<sub>2</sub> as per NASA specification 1080M. Temperature equilibrium in the LN<sub>2</sub> was reached in approximately 35 seconds. After removal from the LN<sub>2</sub> specimens reached room temperature in about 15 to 20 min. All 30 specimens were cycled 10 times with visual inspection after each cycle. All coatings showed good adhesion to the aluminum substrates after 10 immersions in LN<sub>2</sub>. On two of the five MN 2602 samples, a very small amount of coating loss occurred only at the edges of the specimen. Cracking or blistering of the coatings did not occur.

#### **3.3.4. Storage Life**

The storage behavior of replacement coatings for S13G/L0 was investigated. Table 14 lists the results of viscosity tests of coating formulations after six months of storage at -0°F. Little or no change in viscosity occurred for any of the replacement coatings, indicating that no significant cross-linking has occurred.

The coatings were easily redispersed. A light scraping of the bottom of the container was necessary, only to ensure that none of the pigment has compacted beyond the ability of the shaker to break it free from the container. The coating material, which required approximately 5 min for dispersion, showed no sign of lumping, discontinuous pour, or creep. When catalyzed, none of the formulations behaved unusually. There was no rapid increase in viscosity or gelling of the material. Spray characteristics remained very good for all materials. No predeposition polymerization occurred as the material left the spray head, and the resultant coatings were smooth, continuous, and cured in approximately 1 hr.

#### **3.3.5. Space Simulation Studies**

The testing procedure for ultraviolet irradiation in vacuum using the IRIF II was described in Section 3.2.5.1. In the following sections, the results of space simulation studies of pigmented silicone coatings are presented.

TABLE 14. COATING VISCOSITY AFTER 6-MONTH STORAGE AT ROOM TEMPERATURE

Resin		Viscosity, Seconds using No. 4 Ford Cup	
Manufacturer	Design- nation	Initial	After 6 Months
SWS	V-10	25	25
SWS	V-81	25	25
SWS	810	32	35
GE	RTV-12	25	25
MN	R-2602	29	30

#### 3.3.5.1. IRIF II/Test Run 2

The purpose of IRIF II-02 space simulation test was to determine the effects of the UV-vacuum environment on some of the early ZnO pigmented silicone resins formulated on this program. This was also intended as a checkout of the IRIF II system for coating samples. Two samples of each were employed in this test. In addition, 2 samples each of S13G/L0 and Z-93 were included for control purposes, along with an aluminum mirror to aid in the detection of contamination effects as opposed to actual material degradation. In situ reflectance measurements (in vacuo) were made initially (0 ESH) and every 50 hr thereafter (100 ESH) throughout the simulation for 250 hr (500 ESH). Upon completion of each measurement the samples were rotated approximately 30° to minimize any spatial source effects.

The candidate coating formulations used in this simulation all had a pigment to binder ratio (PBR) of 2:1 and were of a thickness range of 6-8 mil. Table 15 summarizes the exposure results, and Figure 30-35 are spectra illustrating the change in absorptance between the initial values and 500 ESH of exposure. The somewhat high initial  $\alpha_s$  values for the experimental coatings are attributed mainly to the nonoptimized PBR's and/or thickness of the coatings.

None of the spectra indicate excessive degradation to the UV vacuum environment. All of the experimental coatings maintain good stability relative to the control S13G/L0. Furthermore, the coatings utilizing SWS V-81 and 810 appear to demonstrate somewhat enhanced stability compared to the S13G/L0.

The lack of a change in UV absorption as a function of time in the spectra of Z-93 and of the aluminum mirror (Figures 34 and 35) also tend to indicate that little if any cross sample contamination occurred during the simulation. Furthermore, this work tends to indicate that only limited amounts of ambient temperature condensables are being leached from these coatings under the conditions of the test. The contaminants that do deposit from these resins seem to demonstrate little if any photodegradation as is evident from the negligible change in the UV absorption of the mirror and Z-93 samples.

TABLE 15. EFFECTS OF VACUUM UV EXPOSURE ON PIGMENTED SILICONE FILMS

Material Designation	Resin	Solar Absorbance Values vs Ultraviolet Exposure (ESH)					$\Delta a$ for 500 ESH	
		0	100	200	300	400		500
S13G/L0 (J-030)	RTV-602/L0	0.17	0.18	0.19	0.20	0.20	0.20	+0.03
I-080	R-2602	0.20	0.22	0.23	0.23	0.23	0.24	+0.04
I-096	V-81	0.21	0.22	0.23	0.23	0.23	0.23	+0.02
I-095	810	0.19	0.20	0.20	0.20	0.21	0.21	+0.02
Z-93 (J-011)	--	0.14	0.14	0.14	0.14	0.13	0.13	-0.01

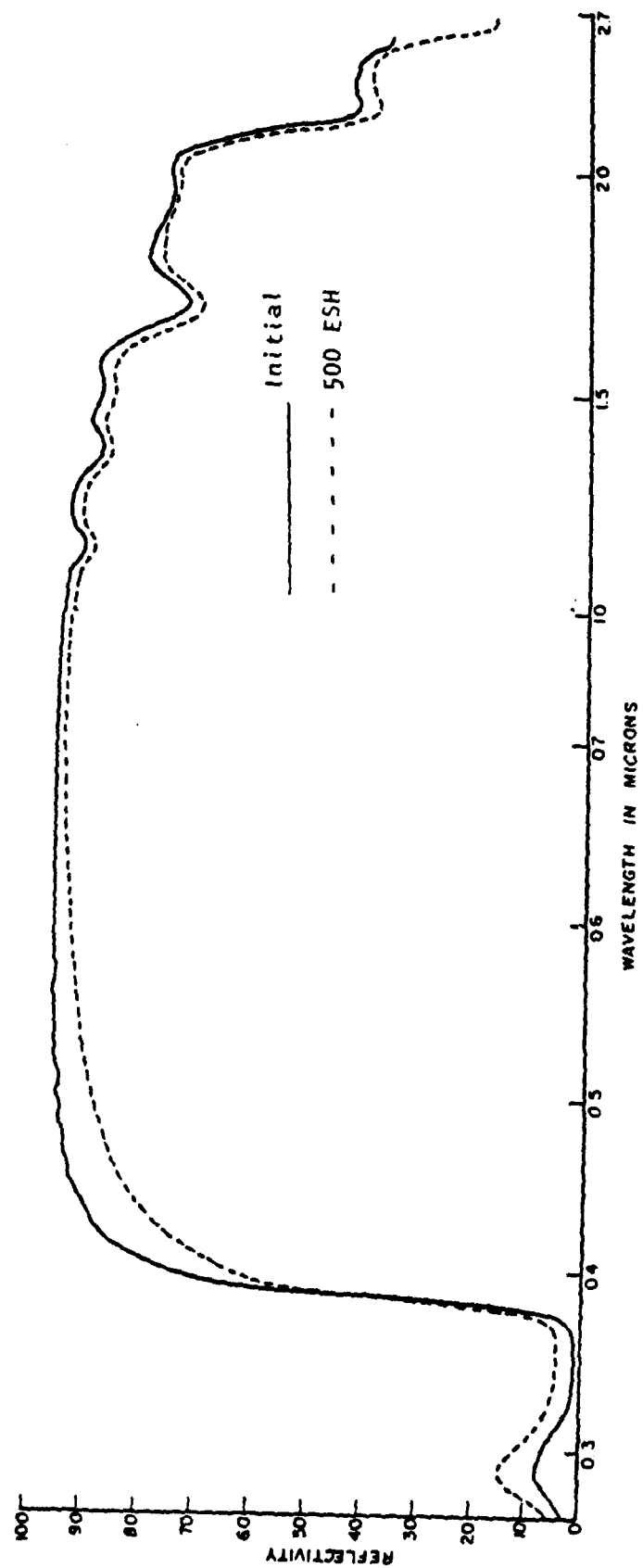


Figure 30. Reflectance spectra of S13G/L0

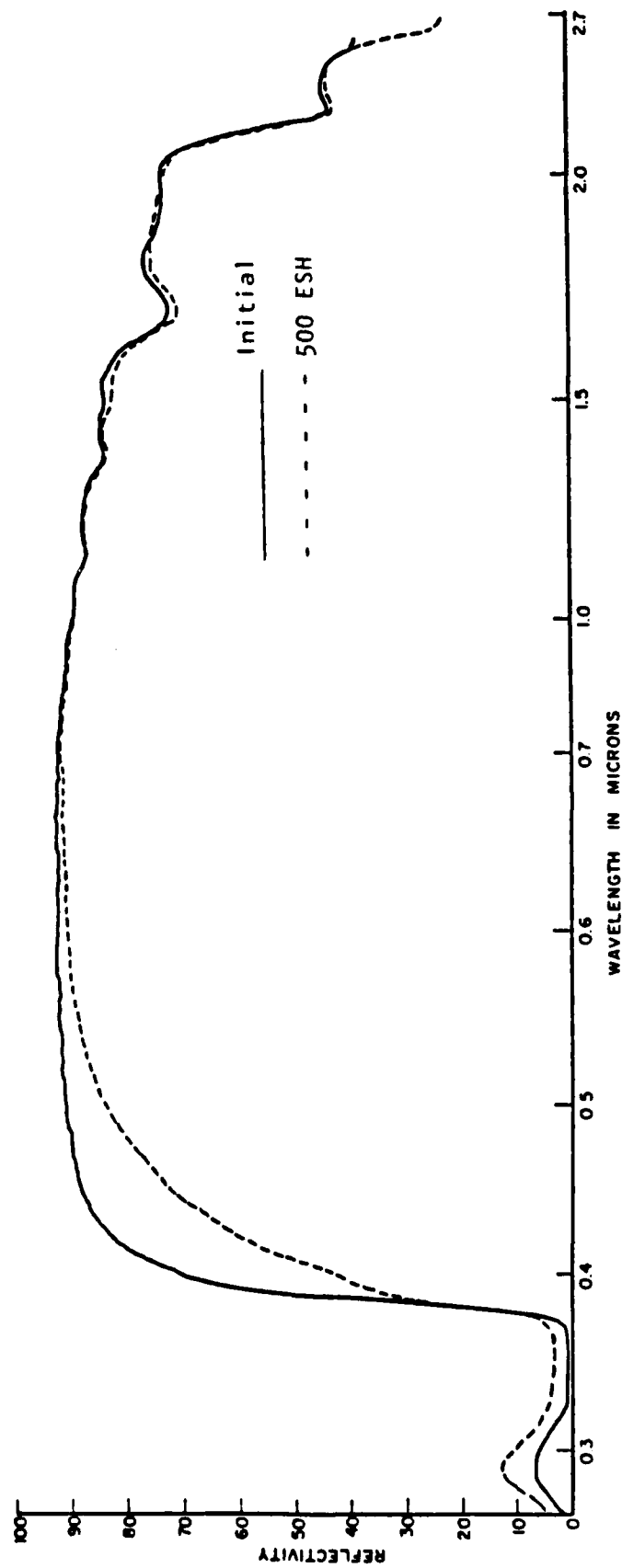


Figure 31. Reflectance spectra of pigmented MN R-2602

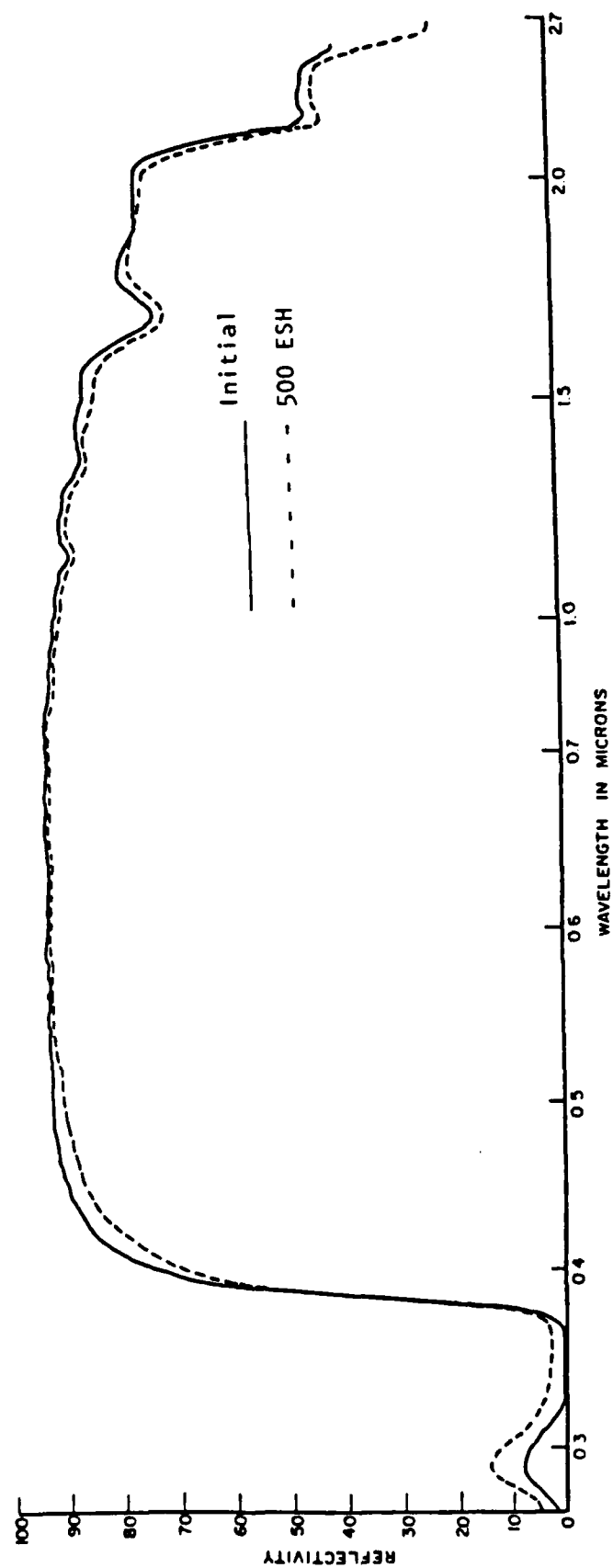


Figure 32. Reflectance spectra of pigmented SWS 810.

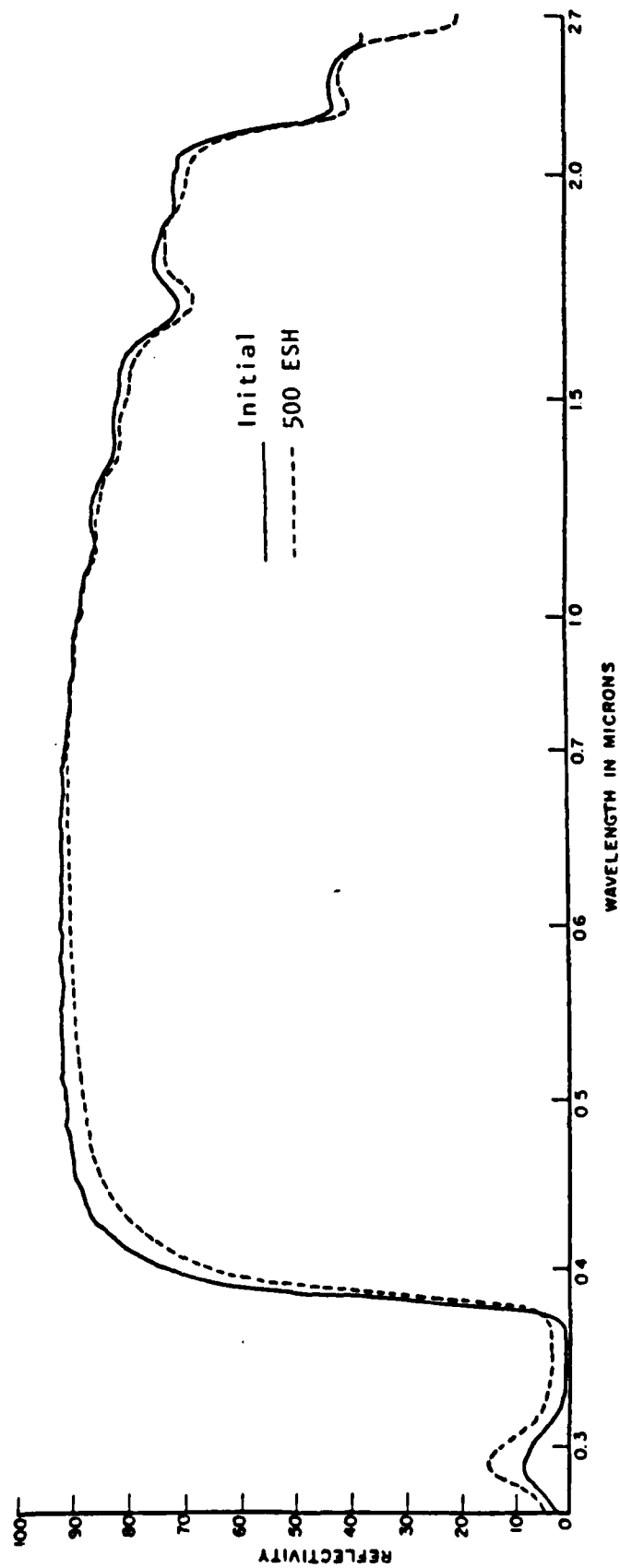


Figure 33. Reflectance spectra of pigmented resin V-81

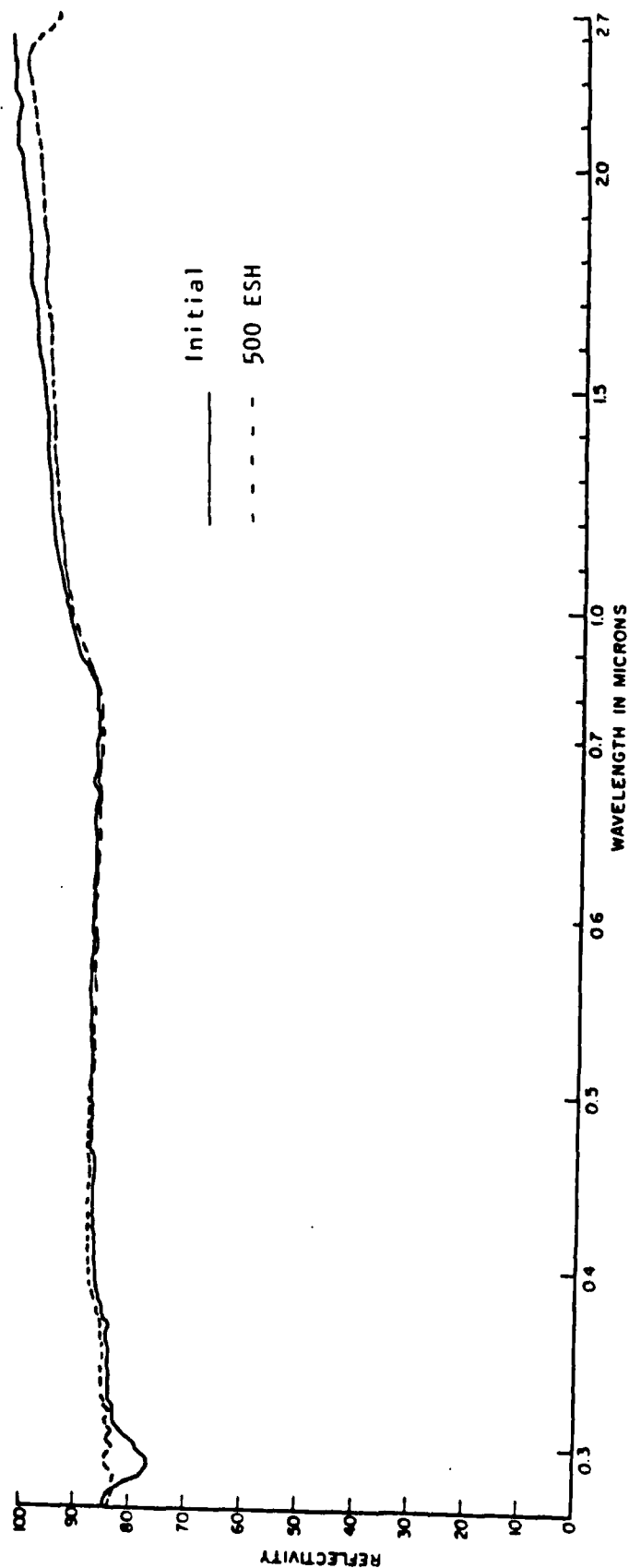


Figure 34. Reflectance spectra of aluminum mirror.

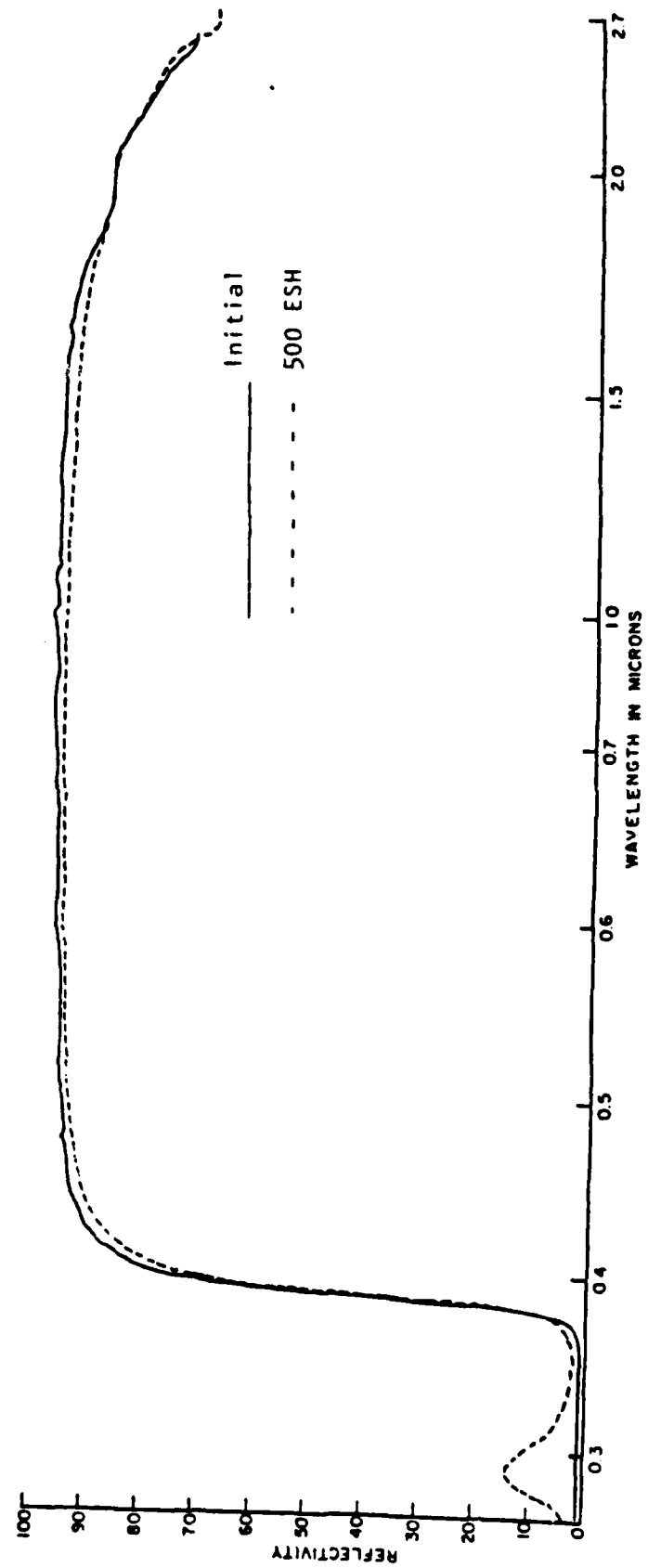


Figure 35. Reflectance spectra of Z-93.

The decrease in  $\alpha_s$  for Z-93 noted in Table 15 is probably related to the volatilization of small amounts of water from the coating, resulting in an increase in infrared reflectance. Also, the reproducibility of these spectra has a range of  $\pm 0.005$ - $0.010$  absorptance or  $\pm 0.5$ - $1.0\%$  reflectance from measurement period to period. This takes into account sample alignment, positioning, and instrumentation noise.

#### 3.3.5.2. IRIF II Run No. 5

The purpose of IRIF II Run No. 5 space simulation was to determine the UV-vacuum effects on three most promising candidate thermal control coating systems produced from the silicone resins listed below:

1. SWS, V-10/L0
2. GE, RTV-12/L0
3. MN, R2602/L0

Samples of S13G/L0 were also included in this test. Three samples of each coating were exposed in this run. All of the coatings were formulated using a pigment-to-binder ratio of 2.1:1. From formulation through irradiation, the materials and samples were handled and treated as identically as possible. Table 16 summarizes sample positions on the irradiation table, the binder, coating thickness, and initial solar absorptance.

The IRIF samples were uniformly coated to a thickness range of 8.0-9.0 mils with the exception of the V-10/L0 samples which have an average thickness of 10.5 mils and one R-2602 sample with a thickness of 9.5 mils. The slightly lower initial  $\alpha_s$  of these samples can be attributed to their greater thickness, but this should have had no effect on their rates of degradation.

Table 17 presents the results from the IRIF II Run No. 5. As is shown in Table 17 and the spectra for the test samples in Figures 36-47 all of the candidate coatings were fairly stable and showed somewhat greater UV-vacuum stability than the reference material S13G/L0 (RTV-602/L0 binder). Some data for the 400 ESH time period is missing because of a spectrometer problem while making the measurements on the samples in-situ. The three samples of each composition behaved quite similarly within the group with a slight variance in solar absorptance ( $\alpha_s$ ) occurring only in one sample of SWS, V-10 (No. 8) and

TABLE 16. COATING SAMPLE POSITION AND THICKNESS

Sample Position	Binder Material	Coating Thickness, mils	Initial Solar Absorptance
1	RTV-602/L0	8.0	0.18
2	RTV-602/L0	8.5	0.18
3	RTV-602/L0	8.0	0.18
4	R2602/L0	9.5	0.17
5	R2602/L0	8.5	0.18
6	R2602/L0	8.5	0.18
7	V-10/L0	10.5	0.17
8	V-10/L0	10.5	0.17
9	V-10/L0	10.5	0.17
10	RTV-12/L0	9.0	0.18
11	RTV-12/L0	9.0	0.18
12	RTV-12/L0	9.0	0.18

TABLE 17. EFFECTS OF UV-VACUUM EXPOSURE ON SILICONE PAINTS COATINGS

No.	Resin Binder Material	Manuf.	Solar Absorption ( $\alpha_s$ ) at Given ESH					Aa after 500 ESH
			0	100	200	300	400	
1	RTV-602/L0	GE	0.18	0.20	0.21	0.22	-	0.23
2	RTV-602/L0	GE	0.18	0.20	0.21	0.22	-	0.23
3	RTV-602/L0	GE	0.18	0.20	0.21	0.22	-	0.23
4	R2602-L0	MN	0.17	0.19	0.20	0.20	-	0.21
5	R2602-L0	MN	0.18	0.19	0.20	0.20	-	0.21
6	R2602-L0	MN	0.18	0.19	0.20	0.20	-	0.21
7	V-10/L0	SWS	0.17	0.18	0.19	0.19	-	0.20
8	V-10/L0	SWS	0.17	0.18	0.19	0.19	-	0.19
9	V-10/L0	SWS	0.17	0.18	0.19	0.19	-	0.20
10	RTV-12/L0	GE	0.18	0.19	0.20	0.20	-	0.21
11	RTV-12/L0	GE	0.18	0.19	0.20	0.20	0.20	0.21
12	RTV-12/L0	GE	0.18	0.19	0.20	0.20	0.20	0.21

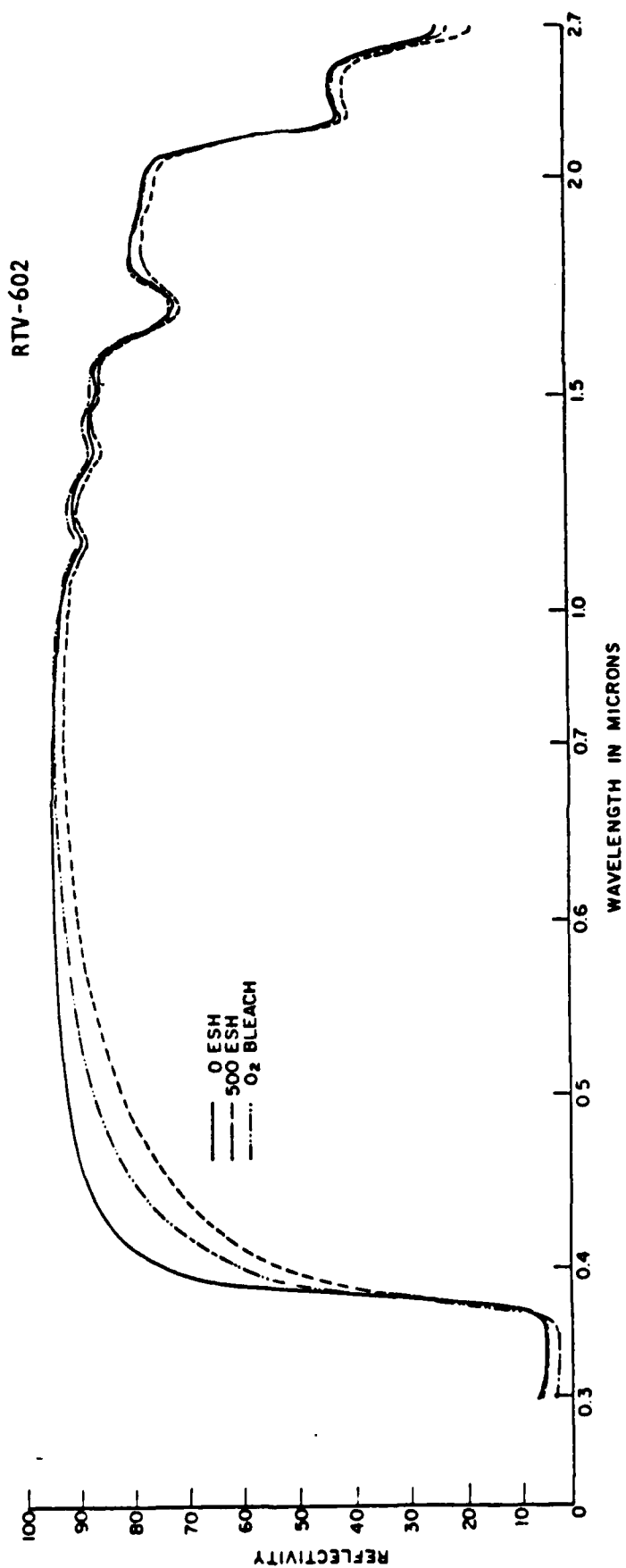


Figure 36. Reflectance spectra of S13G/L0, sample No. 1.

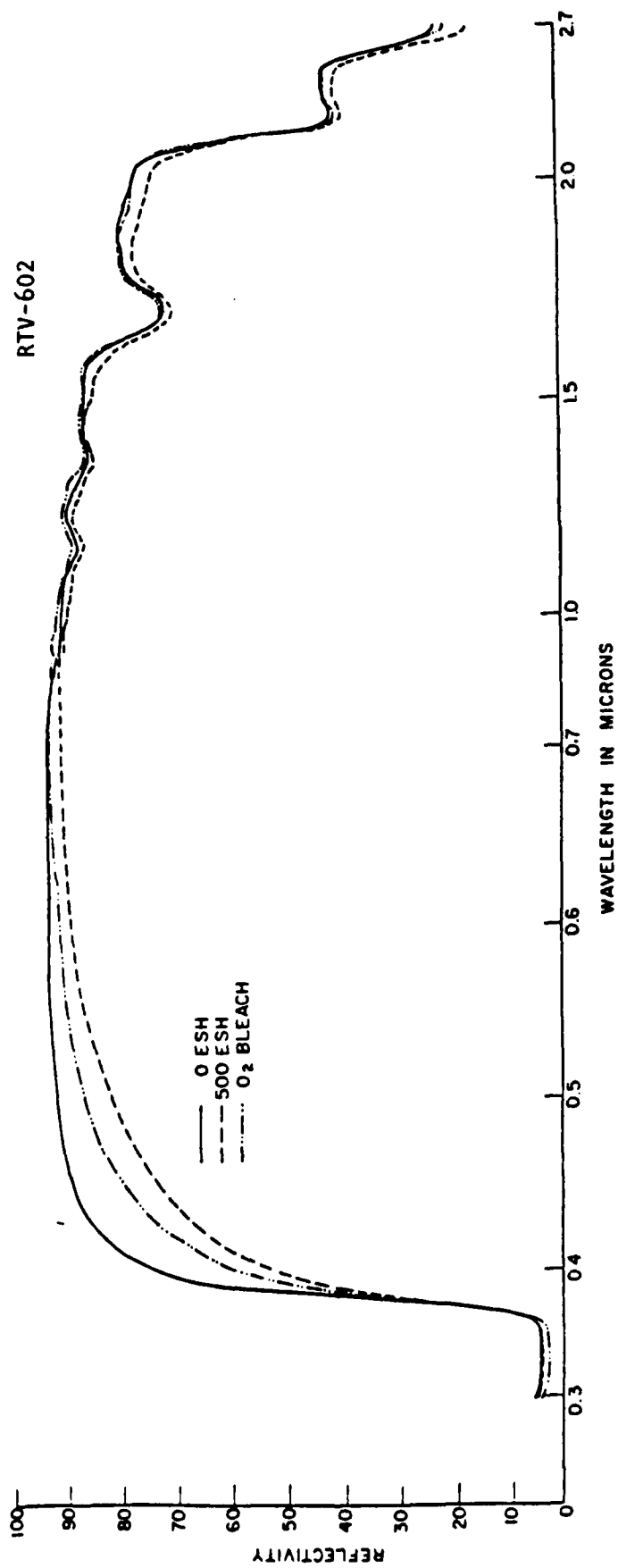


Figure 37. Reflectance spectra of S136/L0, sample No.2

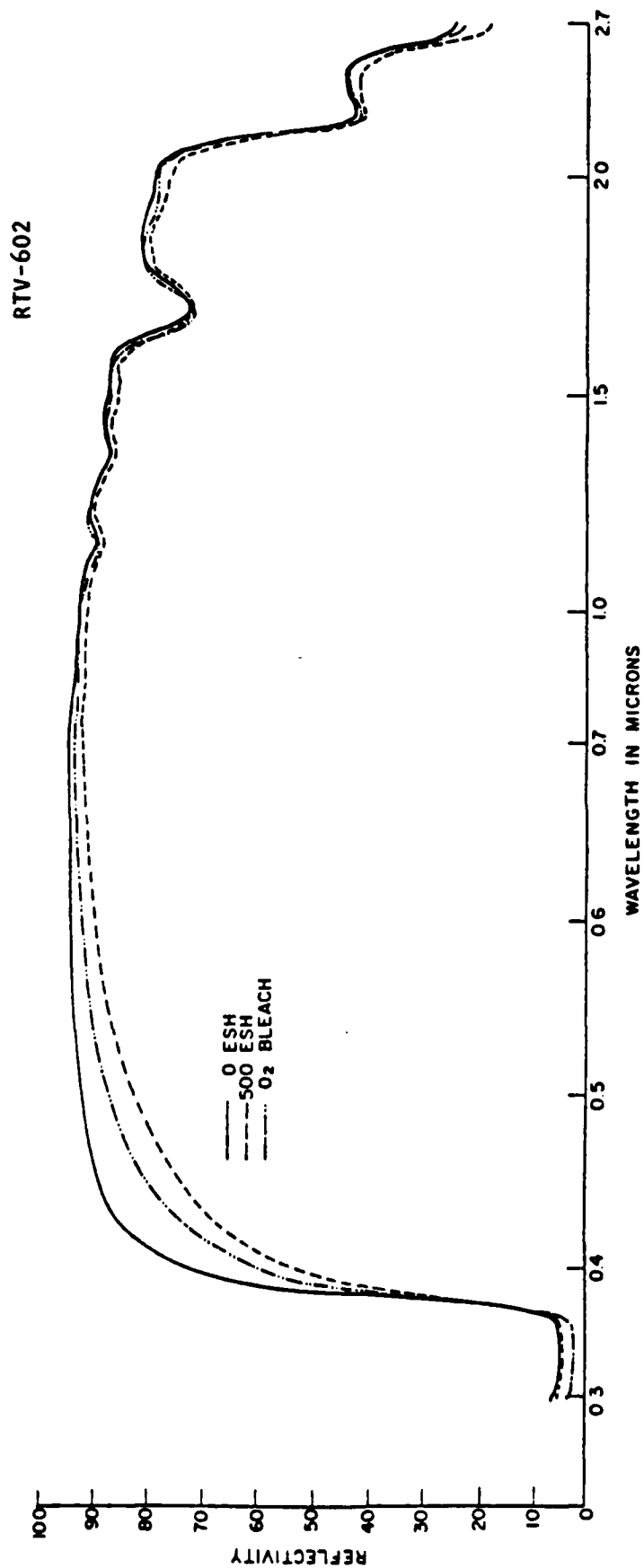


Figure 38. Reflectance spectra of S13G/L0, sample No. 3.

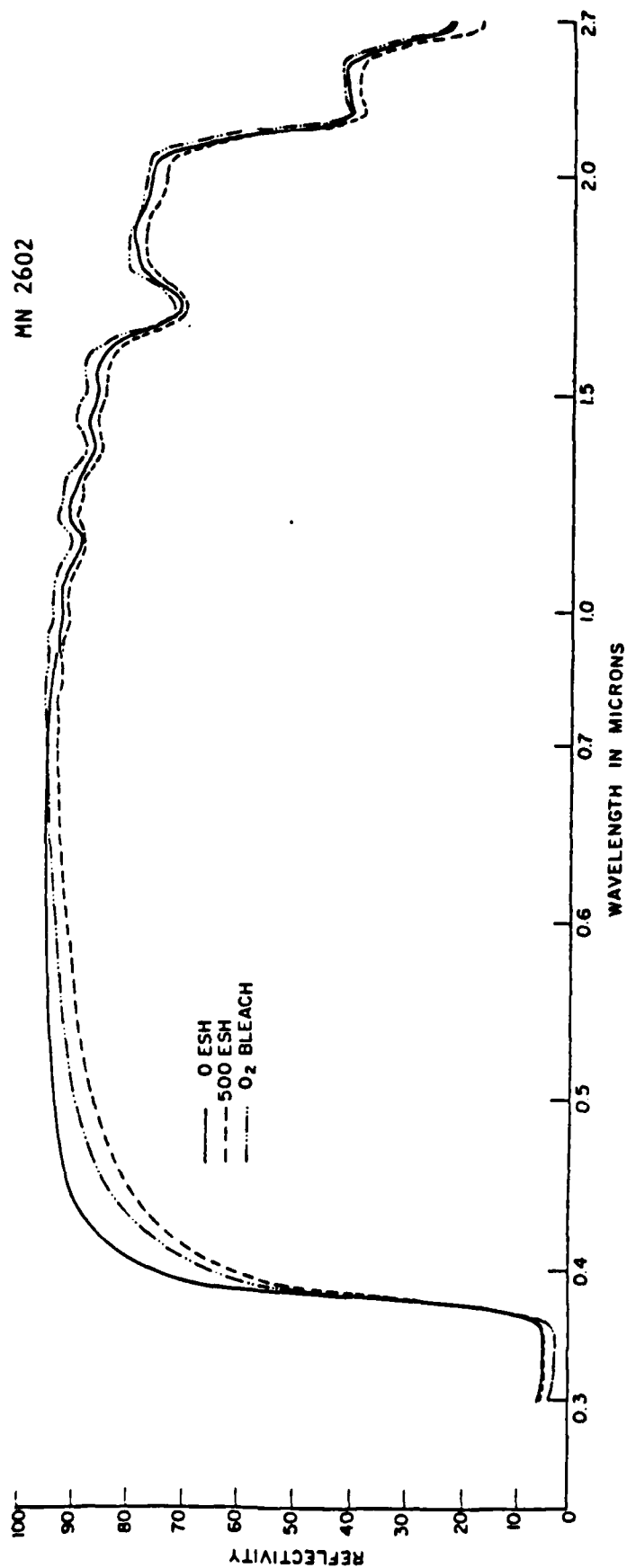


Figure 39. Reflectance spectra of pigmented MN R2602/L0, sample No.1.

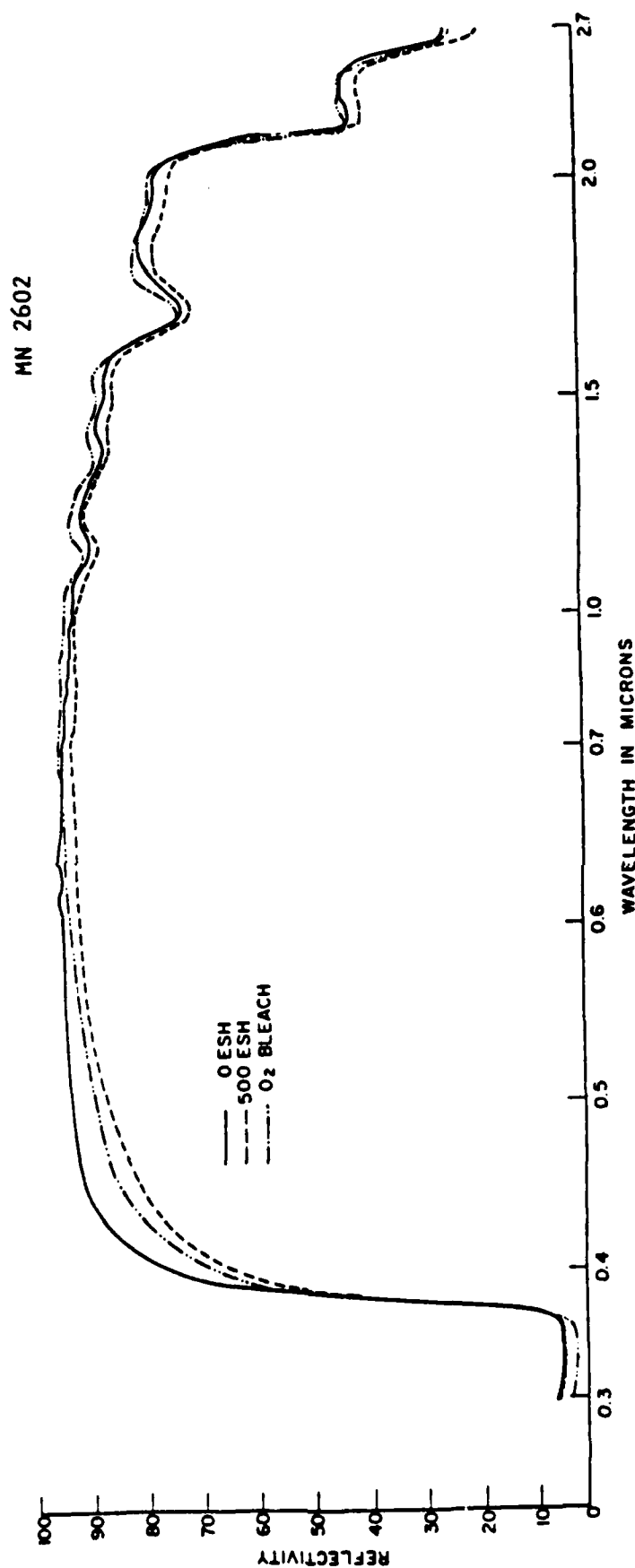


Figure 40. Reflectance spectra of pigmented MN R2602/L0, sample No. 2.

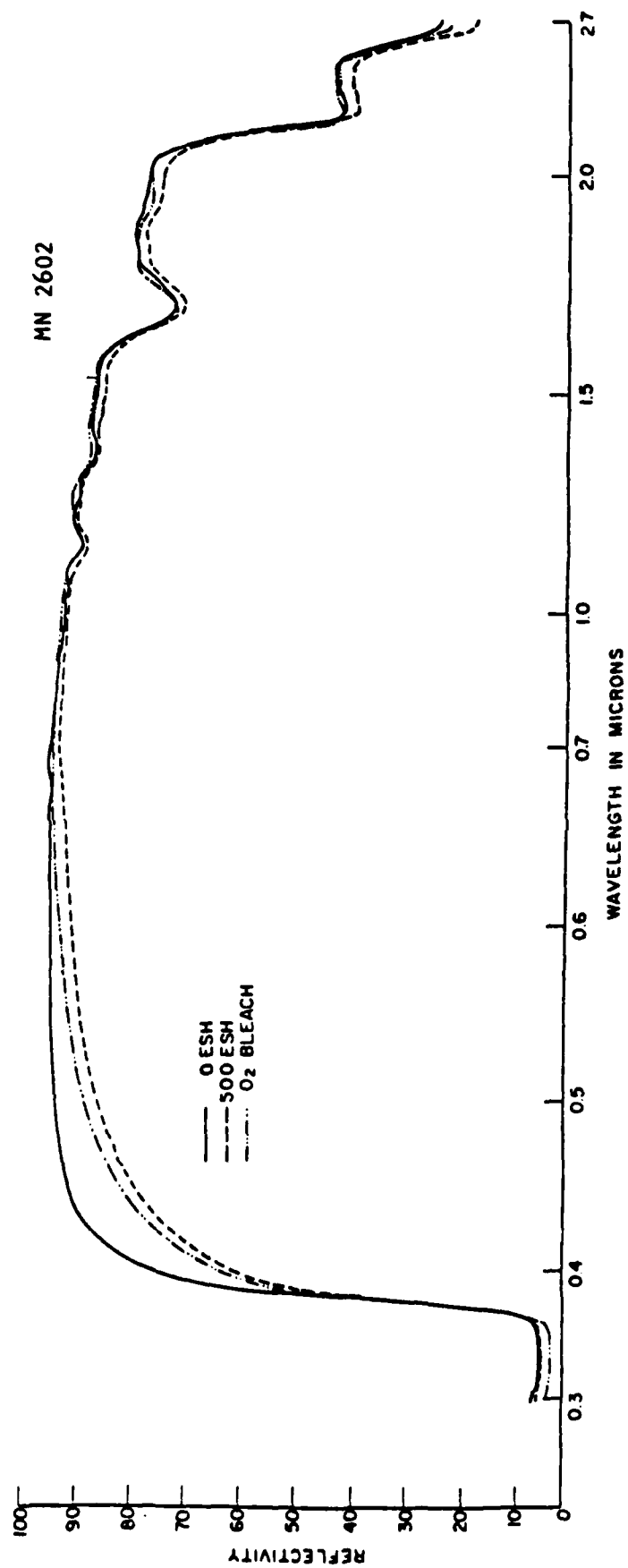


Figure 41. Reflectance spectra of pigmented MN R2602/L0, sample No. 3.

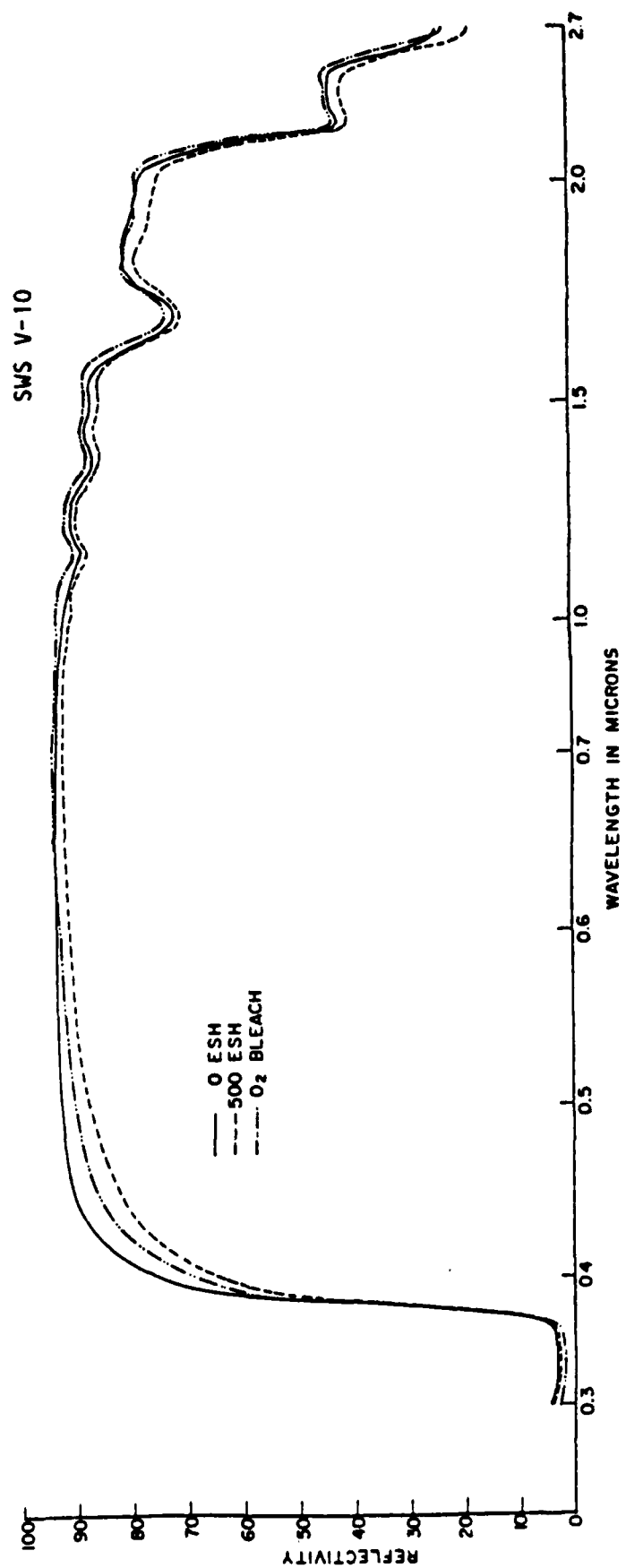


Figure 42. Reflectance spectra of pigmented SWS V-10/L0, sample No. 1.

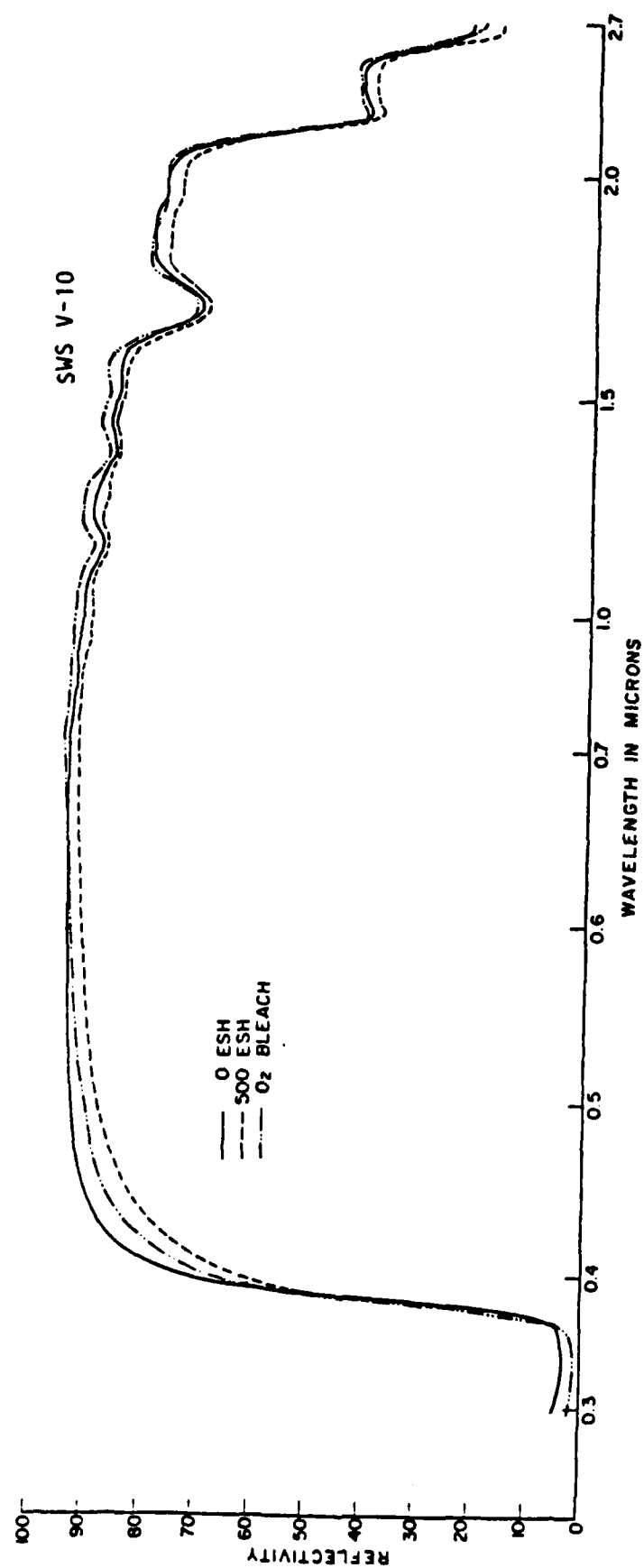


Figure 43. Reflectance spectra of pigmented SWS V-10/L0, sample No. 2.

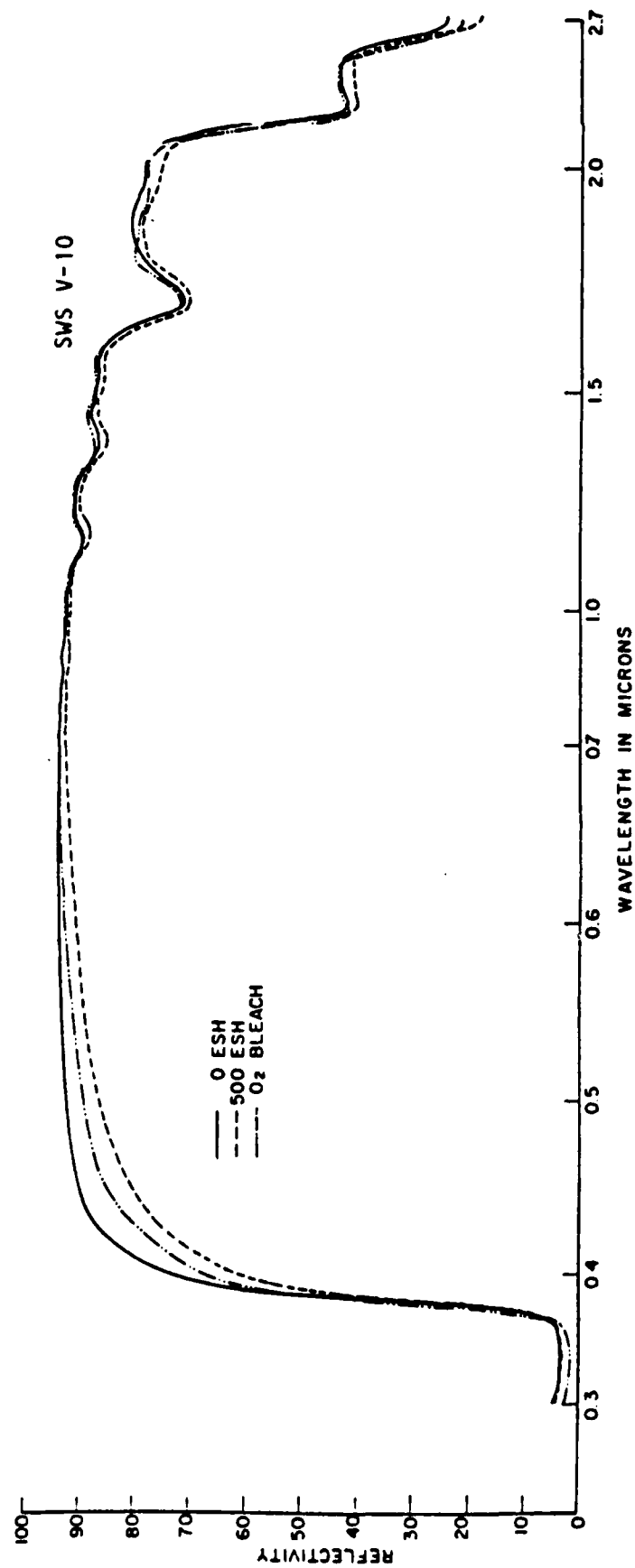


Figure 44. Reflectance spectra of pigmented SWS V-10/L0, sample No. 3.

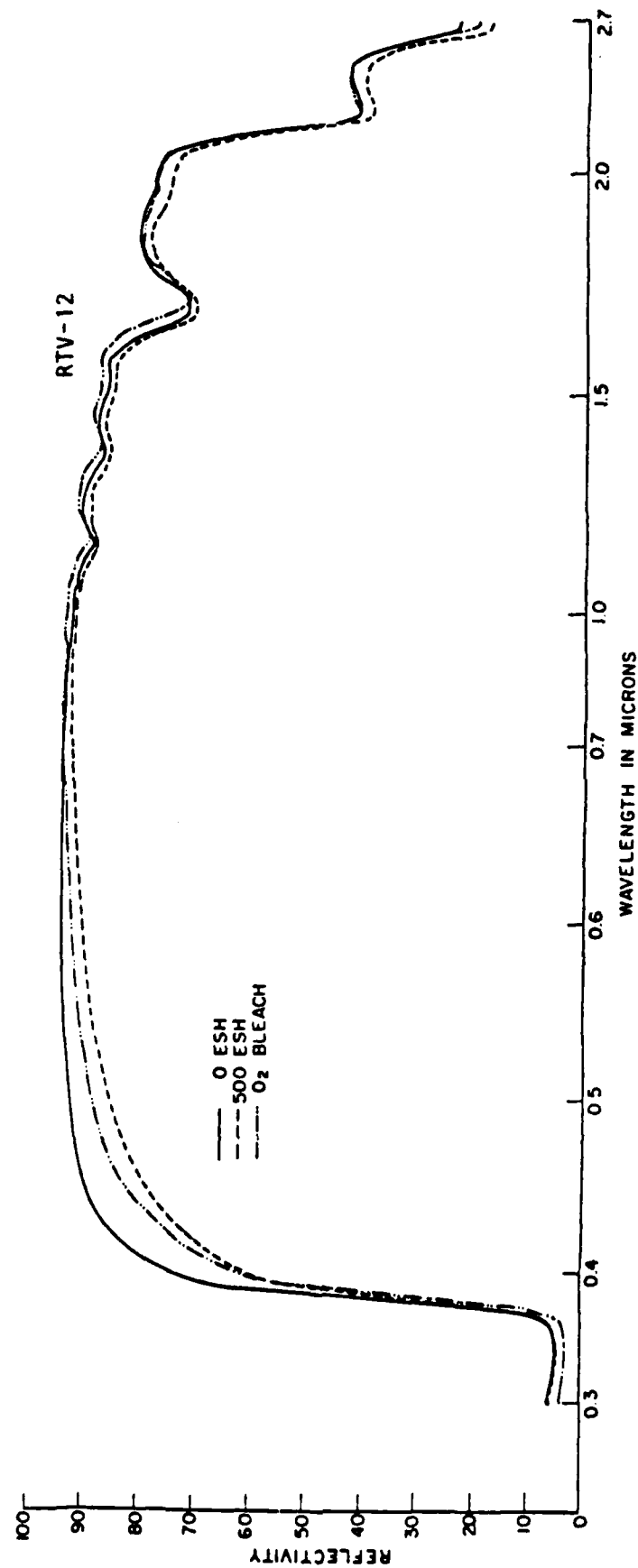


Figure 45. Reflectance spectra of pigmented GE RTV-12/L0, sample No. 1.

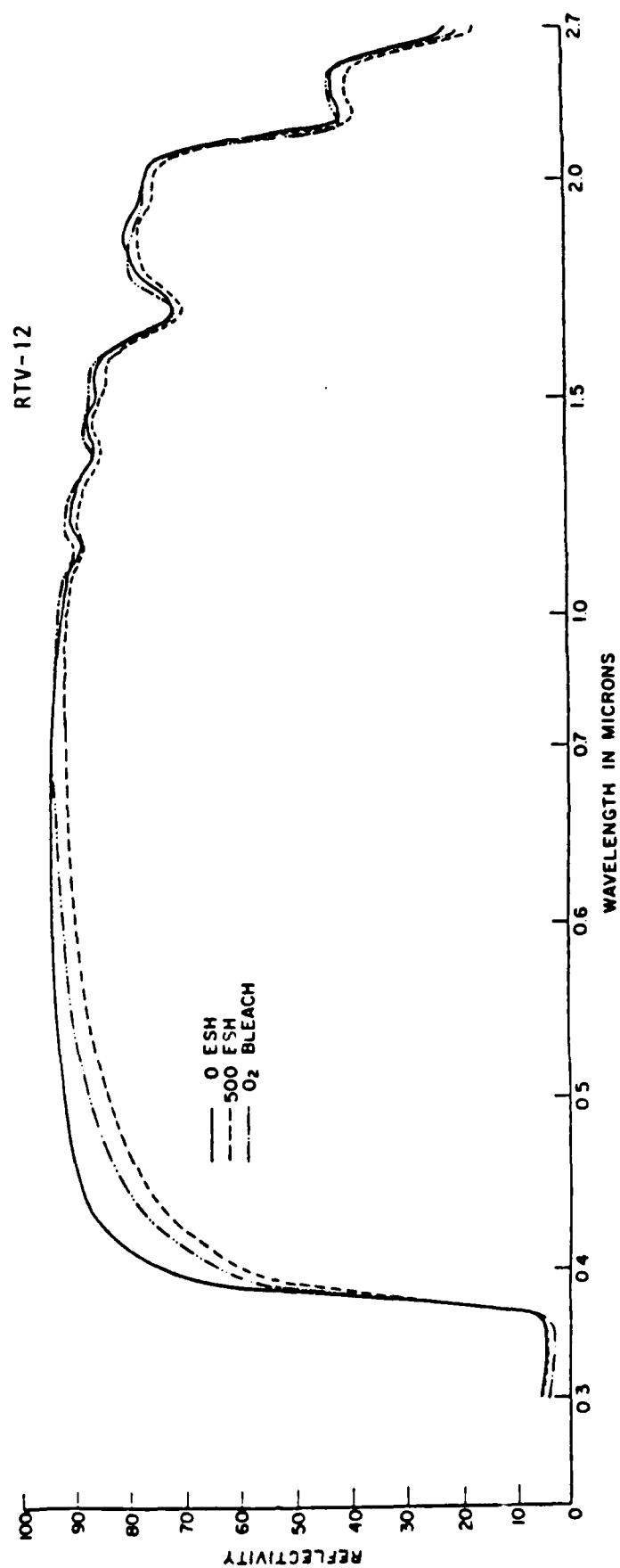


Figure 46. Reflectance spectra of pigmented GE RTV-12/L0, sample No. 2.

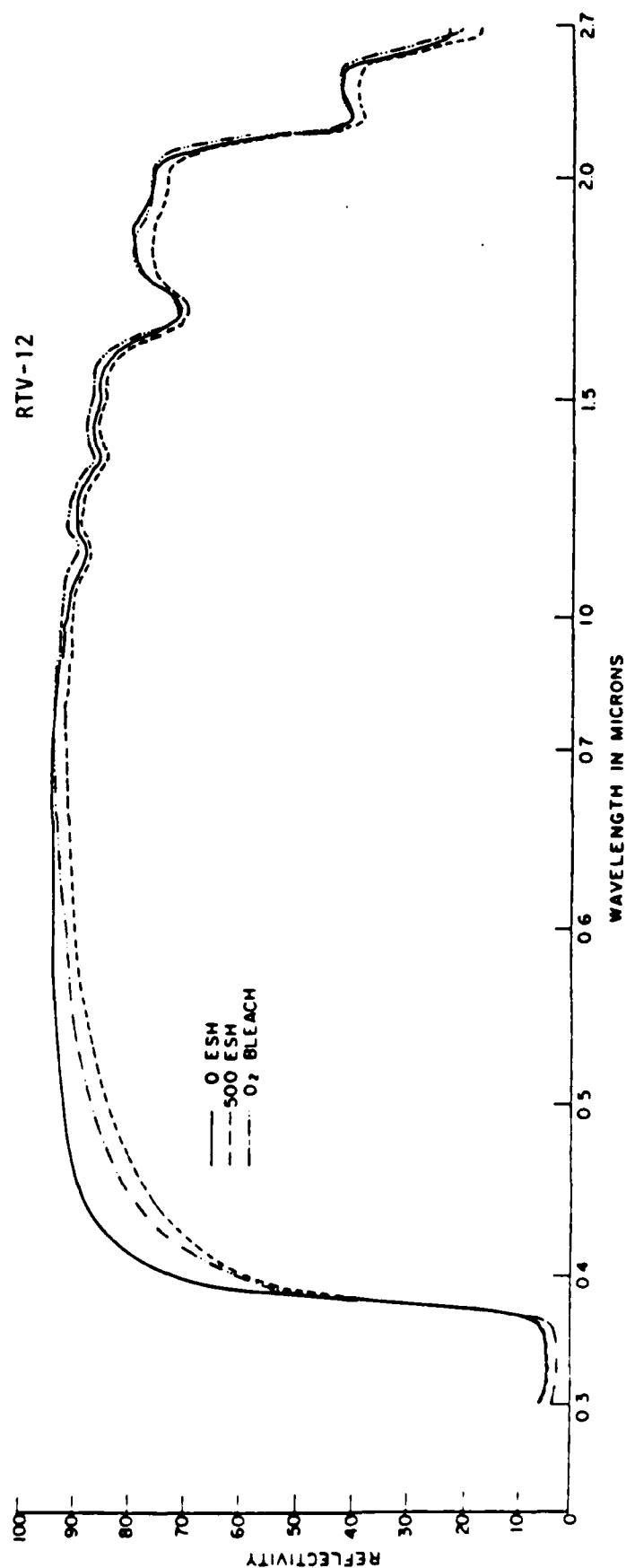


Figure 47. Reflectance spectra of pigmented GE RTV-12/L0, sample No. 3.

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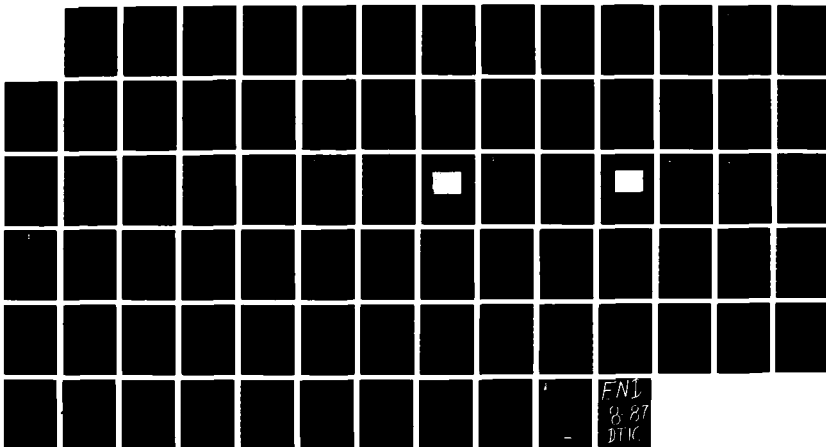
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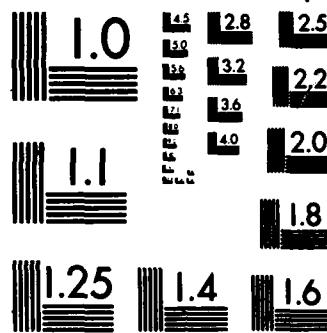
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MN, R-2602 (No. 4). The candidate coatings exhibited an average  $\Delta\alpha_s$  of 0.03 after 500 equivalent sun hours (ESH) which is slightly better than that for S13G/L0 with an average  $\Delta\alpha_s$  of 0.05.

Figure 48 illustrates the rates of degradation for the three candidate coatings and the reference material S13G/L0. As can be seen from this Figure, all three of the candidate coatings have very similar degradation rates. There is a noticeable greater rate of degradation for the S13G/L0 reference as compared to those for the candidate coatings.

All of the coatings tested in Run No. 5 demonstrated a recovery of reflectance when dry purified air was introduced into the vacuum system after 500 ESH of irradiation was completed. As summarized in Table 18, all of the candidate coatings were slightly more effective than S13G/L0 in minimizing the effect of photolytic gas desorption and pigment resin interface interaction, indicated by the lower  $O_2$  bleach  $\Delta\alpha_s$  for the experimental coating. The  $O_2$  bleaching effect in this test required a period of several hours to occur. One of the test samples was monitored from the time air was introduced into the system for a period of one hour, with no noticeable change in reflectance. However, after a period of 24 hours when the samples were remeasured, a measurable reflectance change had occurred. The wavelength of greatest change was at approximately 425 nanometers, with S13G/L0 having the greatest change of 7.0-7.5 percent reflectance increase while the remaining coatings had an increase of 3.0-4.5 percent at the same wavelength. Very little change or recovery occurred in the infrared region of the spectra, indicating that the ZnO in the S13G pigment had not lost, or had its protective overcoating damaged, during mixing and milling of the coating system.

#### 3.3.5.3. Summary

Ultraviolet-vacuum studies at IITRI have shown that formulations consisting of silicated ZnO pigment in SWS V-10/L0, GE RTV-12/L0, or MN 2602/L0, exhibit space environment stability equivalent to or somewhat better than S13G/L0. Excellent stability for the V-10/L0 and RTV-12/L0 coatings was shown in a 2000 ESH test at the Marshall Space Flight Center. The  $\Delta\alpha_s$  was less than 0.03 for the candidate silicone formulations<sup>14</sup>.

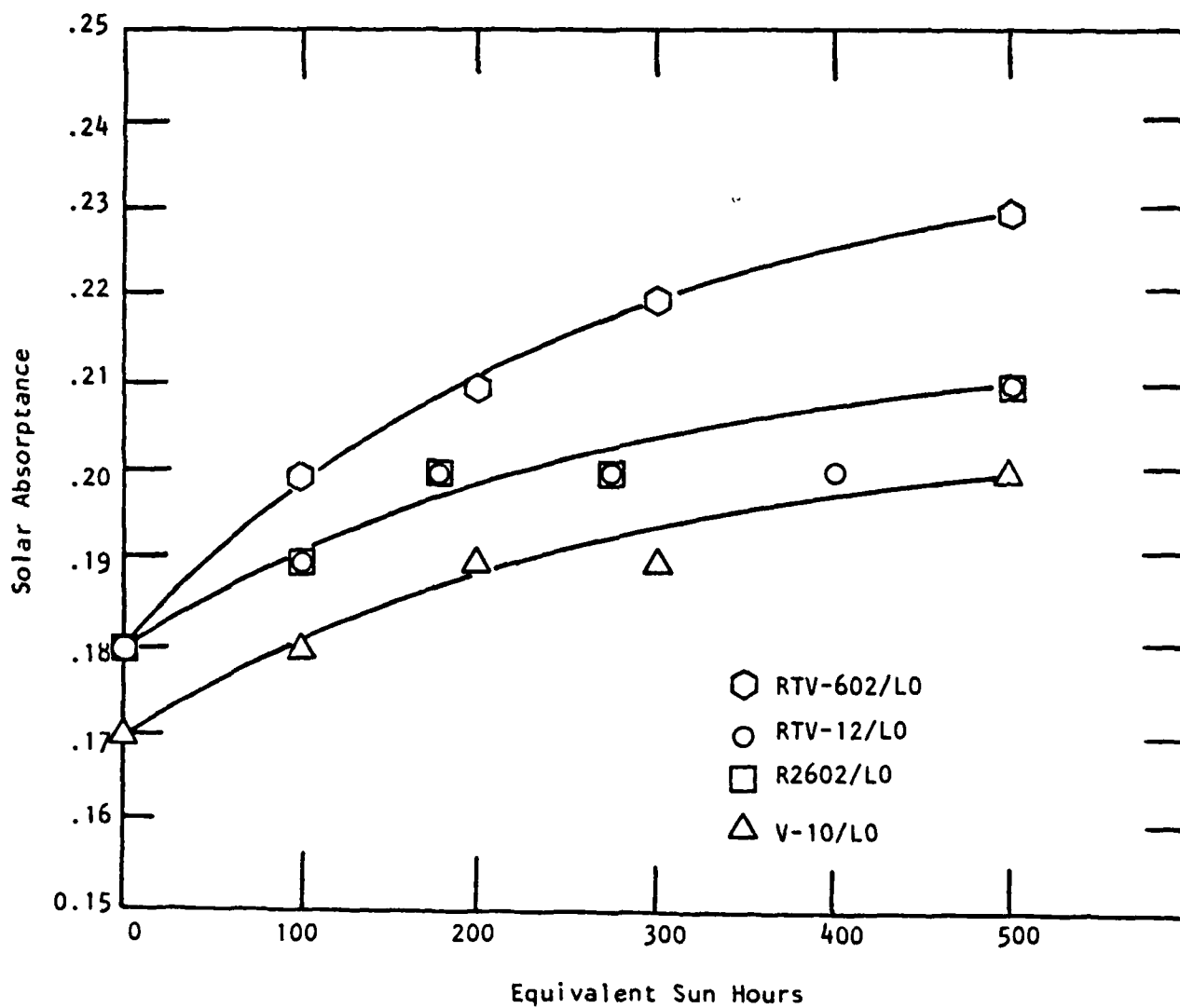


Figure 48. Solar absorbance vs. equivalent sun hours for candidate resin systems.

TABLE 18. REFLECTANCE RECOVERY OF SILICONE PAINTS DUE TO OXYGEN BLEACH

No.	Resin		Initial	Solar Absorptance, $\alpha_s$		O <sub>2</sub> Bleach, $\Delta\alpha_s$	Increase in R after O <sub>2</sub> Bleach at	
	Manf.	Designation		After 500 ESH	After O <sub>2</sub> Bleach		.425 $\mu$	2.0 $\mu$
1	GE	RTV/602/L0	0.18	0.23	0.21	0.02	7.0	1.0
2	GE	RTV/602/L0	0.18	0.23	0.20	0.03	7.5	2.0
3	GE	RTV/602/L0	0.18	0.23	0.21	0.02	7.0	2.0
4	MN	R2602/L0	0.17	0.21	0.19	0.02	3.0	1.5
5	MN	R2602/L0	0.18	0.21	0.19	0.02	3.0	1.0
6	MN	R2602/L0	0.18	0.21	0.21	0.00	3.0	1.5
7	SWS	V-10/L0	0.17	0.20	0.19	0.01	4.5	1.0
8	SWS	V-10/L01	0.17	0.19	0.19	0.00	4.0	0.0
9	SWS	V-10/L0	0.17	0.20	0.19	0.01	4.0	0.0
10	GE	RTV-12/L0	0.18	0.21	0.20	0.01	3.0	1.0
11	GE	RTV-12/L0	0.18	0.21	0.21	0.00	4.0	0.0
12	GE	RTV-12/L0	0.19	0.21	0.21	0.00	4.0	0.0

However, UV-vacuum tests conducted at the University of Dayton Research Institute (Appendix C) showed that the V-10/L0 coating was more stable than coatings using either RTV 602-L0 or RTV-12/L0. Additionally, strong outgassing behavior was exhibited by the RTV-12/L0 material. This behavior indicating incomplete curing was not observed at IITRI or in evaluation at the Marshall Space Flight Center. However, this anomaly must be resolved with additional studies before RTV-12 can be considered a viable backup silicone candidate.

#### 4. CONCLUSIONS

The purpose of this program was to develop a spacecraft thermal control coating which would be a similar if not identical substitute material for S13G/L0. This goal has been met with the development of S13G/L0-1. Key findings in this program are as follows:

1. The SWS V-10/L0 appears to be in the silicone resin best suited as a substitute for GE RTV 602-L0.
2. GE RTV-12/L0 exhibited some serious deficiencies in its outgassing behavior. Additional research should be conducted to identify and resolve some curing anomalies.
3. MN R2602/L0 exhibits good optical properties but has somewhat lower mechanical properties.
4. S13G/L0-1 incorporating SWS V-10/L0 appears to be amenable to scale-up. This is described in the next section.

## 5. SCALE-UP AND DISTRIBUTION

An 8 gallon production batch of S13G/L0 was formulated. This scale-up operation was made with determinations of appropriate milling charge and time, compositional makeup (S13G pigment, SWS V-10/L0, X-99 thinner), and final blending, to achieve the desired formulation viscosity and density. One pint quantities of S13G/L0-1 are currently being distributed to users for their experimentation. IT is anticipated that the results of these various evaluations will be made available to all users of S13G/L0-1.

A distribution list of organizations and personnel who might be interested in S13G/L0-1 was compiled through discussions between IITRI and the Air Force, and through contacts with the aerospace community. This list along with a copy of the distribution inquiry letter appears in Appendix D.

## 6. RECOMMENDATIONS FOR ADDITIONAL WORK

A substitute spacecraft coating, S13G/L0-1, has been developed for use on various vehicles and surfaces for the space environment. Additional work to fully optimize the usefulness of this method should consider full characterization of S13G/L0-1. This will be accomplished in part by the current distribution of sampler amounts and the evaluations to be performed by the various organizations. Studies should also be conducted at IITRI to determine optical, thermal, and electrical properties of S13G/L0-1. In addition, some specific areas of study suggested by the current program are:

1. Full development of backup resin systems, e.g., SWS V-81, GE RTV-615 and GE RTV-12.
2. Use of zinc orthotitanate with new resin systems to obtain lower solar absorptance along with UV-vacuum stability.
3. Investigate the use of fluorosilicones to produce flexible coatings with improved space environment stability.
4. Development of specialty thermal control coatings, e.g., with very high RF transmission for use on antennas.

We further believe that a variety of thermal control coatings having different properties are, and will be needed with longer term and specific mission requirements. Continued research and development can provide materials on a timely basis to meet anticipated needs.

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**APPENDIX A**  
**GEORGE C. MARSHALL SPACE FLIGHT CENTER**  
**NATIONAL AERONAUTICS AND SPACE ADMINISTRATION**  
**MSFC-PROC-1384**  
**S-13G/LO-1 THERMAL CONTROL COATING, APPLICATION OF**



National Aeronautics and  
Space Administration

MSFC-PROC-1384  
9-4-86

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**George C. Marshall Space Flight Center**  
Marshall Space Flight Center, Alabama 35812

S-13G/LO-1 THERMAL CONTROL COATING,  
APPLICATION OF

GEORGE C. MARSHALL SPACE FLIGHT CENTER  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
PROCEDURE

9/11/86  
RELEASE 648

S-13G/LO-1 THERMAL CONTROL COATING, APPLICATION OF

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GEORGE C. MARSHALL SPACE FLIGHT CENTER  
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
PROCEDURE

S-13G/LO-1 THERMAL CONTROL COATING, APPLICATION OF

1. SCOPE

1.1 Scope. This specification covers the detail requirements for S-13G/LO-1 thermal control coating and its application on space flight hardware.

1.2 Classification. The process covered by this specification consists of the following classes:

- (a) Class I - This class is designated for spacecraft hardware with simple configuration where strict control of the coating thickness is not difficult.
- (b) Class II - This class is designated for spacecraft hardware with a complex configuration where strict control of the coating thickness is not practical.

2. APPLICABLE DOCUMENTS

2.1 The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposals shall apply.

SPECIFICATIONS

Federal

BB-N-411	Nitrogen, Research grade
TT-T-548	Toluene, Research grade
O-E-760	Ethyl Alcohol (Ethanol); Denatured Alcohol; and Proprietary Solvent.

Military

MIL-A-8625	Anodic Coatings, for Aluminum and Aluminum Alloys.
MIL-P-17667	Paper, Wrapping, Chemically Neutral (Non-Corrosive).
MIL-C-26074	Coating, Nickel-Phosphorus, Electroless Nickel.
MIL-M-3171	Magnesium Alloys, Process for Corrosion Protection of.
MIL-M-45202	Magnesium Alloys, Anodic Treatment of.
MIL-STD-171	Finishing of Wood and Metal Surfaces.
MIL-C-5541	Chemicals, Films and Film Materials, for Aluminum and Aluminum Alloys.

American Society of Testing Materials

ASTM-E408-71	Total Normal Emittance of Surface Using Inspection Meter Techniques.
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Lyndon B. Johnson Space Center

SP-R-0022A	General Specification - Vacuum Stability Requirements of Polymeric Materials for Spacecraft Applications.
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(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

### 3. REQUIREMENTS

3.1 General Considerations. S-13G/LO-1 thermal control paint is a dispersion of potassium silicate-treated zinc oxide (ZnO) pigment in a specially prepared silicone elastomer vehicle with volatile solvents employed as required.

3.2 Qualification. The paint furnished under this specification shall be a product that has been tested and has passed the qualification tests specified herein. Any change in the

formulation of a qualified product will necessitate its requalification. Paint supplied under contract shall be identical, within manufacturing tolerances, to the product receiving qualification.

3.3 Properties. The following requirements apply to paint supplied by the manufacturer.

3.3.1 Thermal Emittance. The total normal emittance of the coating shall be  $0.90 \pm 0.05$  measured with a Gier-Dunkle DB100 infrared reflectometer or equivalent.

3.3.2 Solar Absorptance. The solar absorptance of the coating shall be not greater than 0.22 when measured with a Gier-Dunkle Integrating Sphere Reflectometer (or approved equal) in the wavelength range of 325 to 2600 nanometers, at ambient room pressure and temperature.

3.3.3 Ultraviolet-Vacuum Degradation Rate. The change in solar absorptance of the coating shall not be greater than 0.05 after 1000 equivalent sun hours.

Note: The stability of S-13G/LO-1 in the ultraviolet (UV)-vacuum degradation test is highly sensitive to the effects of minor contaminants. Care must be exercised in all paint preparation and application operations to maintain the requisite cleanliness. All equipment used must be kept clean.

3.3.3.1 Initial Solar Absorptance. The initial solar absorptance for the degradation rate test shall be determined using a Gier-Dunkle Integrating Sphere Reflectometer, or equivalent, in a wavelength range of 325 nm to 2600 nm at a maximum pressure of  $10^{-7}$  torr and a temperature of  $75 \pm 5$  °F.

3.3.3.2 Ultraviolet Exposure. The sample shall be exposed to continuous radiation from an Xenon arc, mercury-xenon arc, or equivalent source, for a period of 500 equivalent sun hours. The irradiance per equivalent sun hour shall be 13 milliwatts per square centimeter in the wavelength region below 400 nm. The sample shall be maintained at a maximum pressure of  $10^{-7}$  torr and a temperature of  $80 \pm 10$  °F during this exposure.

3.3.3.3 Degradation. The solar absorptance shall again be measured as in 3.3.3.1. The sample shall be maintained at a maximum pressure of  $10^{-7}$  torr between the time of the end of the ultraviolet exposure of 3.3.3.2 and the time of this measurement. The difference in initial and degraded solar absorptance shall be reported as the change in solar absorptance.

3.3.4 Adhesion. The coating covered by this specification shall not exhibit loss of adhesion to aluminum when tested in the following manner. Two coated test samples shall be thermally shocked by immersion in liquid nitrogen. Samples shall remain immersed until thermal equilibrium with the liquid nitrogen is established and violent boiling ceases. The samples shall then be removed from the liquid nitrogen and allowed to return to room temperature. The coatings shall then be cut with a knife blade inserted into the cut and turned parallel to the substrate. Cutting, crumbling, or mechanical failure of the coating other than by lifting of the film shall not constitute loss of adhesion.

Note: The coating is not exceedingly tough. Any attempt to mechanically remove the entire film may result in cohesive failure of the coating; however, where adhesion is adequate the film will not lift or strip in large pieces.

3.3.5 Spraying Properties. The paint shall spray satisfactorily in all respects and shall show no running, sagging, or streaking. The cured film shall show no dusting or mottling and shall present a smooth uniform finish free from seediness; however, an egg-shell or dull stipple effect or low gloss shall be considered normal for the coating.

3.3.6 Weight. The weight of the paint covered by this specification shall be 11.9 to 12.5 pounds per gallon.

3.3.7 Viscosity. The viscosity of the paint covered by this specification shall be within the range of 30 to 37 seconds when tested with a No. 2 Zahn cup or 25 to 31 seconds using a No. 4 Ford cup. This viscosity requirement shall apply only at the time of manufacture of the paint.

3.3.8. Pot Life. The pot life of the catalyzed paint covered by this specification shall be at least one hour. Any catalyzed paint not used within one hour shall be discarded.

3.3.9 S-13G/LO-1 Shelf Life. The shelf life of the paint shall be guaranteed by the supplier for a period of 60 days following the date of receipt by the procuring activity provided that the paint is stored at a temperature of 42 degrees Fahrenheit (°F) or less. During the guaranteed shelf life period, the supplier shall warrant that the viscosity of the paint will not exceed 70 seconds using a No. 4 Ford cup and at a temperature between 72 and 78 °F. After the expiration of the guaranteed shelf life period, the paint may be used until the viscosity exceeds 100 seconds using a No. 4 Ford cup or the paint begins to gel.

3.3.10 Catalyst Shelf Life. The shelf life of undiluted catalyst shall be 6 months from the date of shipment by the catalyst supplier. Storage requirements of 3.3.9 shall be observed.

3.3.11 Thermal Vacuum Stability. Following a cure of 21 days at room temperature, the coating shall meet the total weight loss and vacuum condensible material requirements of SP-R-0022A.

3.3.12 Toxicity. When used for its intended purpose and applied in accordance with specification, the paint shall have no adverse effect on the health of personnel.

3.4 Process Control. Process controls shall be exercised in accordance with the following paragraphs.

3.4.1 Work Order Form. A work order form or other suitable document shall be used to provide instructions to the operator, to record compliance to the requirements specified herein, and to achieve traceability. The form of document shall be completely filled in, including hardware serial numbers and spacecraft models. Special handling, control, or precautions shall be noted on the form or document. The work order form or other suitable document shall also state the cure requirements and/or limitations for the hardware item(s) to be processed, so that the proper manufacturer's lots can be selected to make up the paint batch per 3.4.2.

3.4.2 Batch Identification and Control. For the purpose of this specification, a batch of paint shall be considered as that supply of paint prepared in one operation. All paint used to make up a batch shall have passed the acceptance requirements. The identifying numbers or codes assigned by the manufacturer to all paint in the batch shall be recorded to maintain traceability. Unless otherwise specified, control samples shall be prepared, during the processing of parts, in accordance with 3.4.2.1. These samples shall be identified with the processed parts. Care shall be taken to assure that the manufacturer's lot(s) selected for the batch is approved for the cure appropriate to the hardware item processed.

3.4.2.1 Production Control Sample Preparation. Production control samples for adhesion, thermal emittance, and solar absorptivity, shall be prepared concurrently with each paint application cycle. Control sample substrate shall be the same type material as the hardware with a minimum thickness of 0.020-inch, and number and size shown in Table I unless otherwise required by the test equipment to be utilized. Surface preparation, priming, coating, and curing of the control samples shall be done in the same manner and at the same time as the hardware.

TABLE I. BATCH CONTROL SAMPLES

Test	Number of Samples	Substrate Size
Adhesion	3	2"x2"
Thermal Emittance	3	7/8" diameter
Solar Absorptance	3	7/8" diameter

3.4.3 Thickness. Coating thickness shall be  $8.0 \pm 2.0$ , -1.0 mils. Thickness measurements for Class I and Class II processes shall be made as follows:

- (a) Class I. Class I thickness measurements shall be made directly on the work part, after the paint has been applied and cured, using a standard thickness gauge measuring device such as capacitance or eddy type and the measuring procedure specified in 4.2.3.
- (b) Class II. Class II thickness measurements for control of the paint spraying process, independent of the configuration of parts, shall be made on panel specimens 6x6 inches. These specimens shall be painted in the same spray application cycle and shall be cured in the same cycle along side painting operations applied to the work part.

3.4.4 Hardware Release. Once a part has been submitted for processing, it shall not be released until it has completed the curing process specified in 3.6.4.

3.4.5 Protection of Parts. During and after any operation specified herein, parts shall be handled in a manner that will minimize biorganic substances, dust, or other foreign matter from contaminating the surfaces being processed. Protective coverings of Tedlar, Teflon, or Kynar films may be used. For the purpose of this specification, fingerprints, perspiration, dandruff, hair follicles, and saliva shall be considered biorganic contaminants. In all cases, flight hardware shall be protected from random temperature extremes, high humidity, contamination, and physical damage. The temperature control paint shall not be applied prior to any mechanical operations such as machining, drilling, forming, or welding. Adhesive bonding on surfaces to be painted shall have been completed prior to paint application.

3.5 Facilities Requirements. The temperature control paint shall be applied in paint room facility, specifically designated to serve as a thermal control paint application facility and reserved for that purpose, to assure that the applied coating meets the requirements specified herein. Surface preparation, precleaning, inspection, storage, and operations subordinate to paint application shall be performed in an area adequately controlled to assure compliance with the requirements specified herein. The environmental conditions of the paint facility, used for the process specified herein, shall be in accordance with 3.5.1 through 3.5.6.

3.5.1 Temperature. The paint facility shall be capable of maintaining the temperature at 75  $\pm$  15 °F during the processing of parts.

3.5.2 Humidity-Temperature. Temperature of the parts to be coated shall not be allowed to fall below the dewpoint of the ambient air. If condensation on parts in process is observed, paint operations shall be immediately suspended. In the event that the relative humidity of the paint facility exceeds 50 percent, parts that have been prepared for painting by the alkaline etch or mechanical surface treatments of 3.6.1.1 shall be processed as rapidly as possible. In no event shall etched or mechanically cleaned surfaces be exposed to high humidity conditions prior to priming for a period of time greater than 24 hours.

3.5.3 Intake Air Filters. All intake air to the paint room facility shall be drawn through filters having a minimum efficiency of 30 percent on the NBS atmospheric dust test. Filter elements shall be changed with sufficient frequency to assure efficient dust removal.

3.5.4 Air Flow. The velocity of the air flow at the spray table in the paint booth shall be within the range of 100 to 150 feet per minute and shall be sufficient to prevent dry overspray from settling on surfaces which have been painted and are still tacky.

3.5.5 Vapor Control. The vapor from solvents shall be controlled by means of a positive exhaust at the rear of the paint spray booth. Vapor handling and protection of the operator from solvent fumes shall be in accordance with applicable industrial hygiene safety regulations.

3.5.6. Personnel Protective Precautions. All precautions for personnel protection shall be observed to prevent the inhalation of fumes or dust generated by vapor degreasing, surface treatment, and painting.

**3.6 Temperature Control Painting Process Requirements.** The standard process for the application of temperature control paint shall consist of surface preparation by chemical or mechanical methods, cleaning, masking of areas to be free of paint, paint application, and special handling of parts. Class I and Class II temperature control paint application processes shall be performed as follows:

- (a) Class I. Parts designated for Class I temperature control paint application shall be processed in accordance with 3.6.1 through 3.6.7.
- (b) Class II. Parts designated for Class II temperature control paint application shall be processed in accordance with 3.6.1 through 3.6.7. A coating thickness sample representative of the part shall be processed in conjunction with the processing of the part as specified in 3.4.3(b).

**3.6.1 Surface Preparation.** Surface preparation, to promote adhesion of the temperature control coating, shall consist of either a chemical or mechanical process designed to produce an effective surface finish on the substrate. Surfaces shall be cleaned and prepared in accordance with the procedures specified in the following paragraphs.

**3.6.1.1 Aluminum.** Parts that are constructed of aluminum alloys shall be cleaned by one of the following methods:

- (a) Mechanical Surface Preparation
  - (1) Abrade with 120 to 320 grit aloxite cloth or paper, Scotch-Brite abrasive pads, or dry grit blast with 100 to 150 grit aluminum oxide.
  - (2) Solvent wipe with ethyl alcohol conforming to Specification O-E-760, Grade III and blot dry with lint-free cloth or wipes. Allow surface to dry for at least 1 hour before application of primer.
- (b) Anodize as specified in Specification MIL-A-8625 for Type I, Class I, unsealed.

(c) Alkaline Etch

- (1) Vapor degrease
- (2) Etch in a 5 percent solution of sodium hydroxide (NaOH) for 3 minutes at room temperature
- (3) Rinse with water
- (4) Desmut in a 15 percent solution of nitric acid
- (5) Rinse with water and dry.

Note: Parts or assemblies that are adhesively bonded or that include polymeric or organic materials in their construction shall not be vapor degreased. Metal surfaces shall be cleaned by solvent wiping after any specified areas have been protected by masking.

- (d) Chemical conversion coat per Specification MIL-C-5541, Iridite 14-2 or Alodine 1200 only, and prime per 3.6.1.5.

3.6.1.2 Fiberglass Plastics. Fiberglass plastic surfaces shall be cleaned as follows:

- (a) Scrub with a 10 percent solution of Alconox (or approved equal)
- (b) Roughen with 180-240 Aloxite cloth (or approved equal)
- (c) Wash with water and dry
- (d) Scrub with 95 percent ethyl alcohol, Specification O-E-760, Grade III
- (e) Rinse with 95 percent ethyl alcohol and dry.

3.6.1.3 Magnesium Alloys. Magnesium alloys shall be prepared by one of the following methods:

- (a) Iridite per Specification MIL-M-3171C, Type VIII, and prime per 3.6.1.5.
- (b) Chemical conversion coat with Iridite 15 or per Specification MIL-M-3171 (Dow 7) and prime per 3.6.1.5.

3.6.1.4 Other Metals and Alloys. The following surface preparations shall be used for the listed alloy types:

- (a) Corrosion resistant steel alloys - Passivate per 5.4.1 of MIL-STD-171.
- (b) Titanium alloys - Clean per 4.3 of MIL-STD-171.
- (c) 4130 steel alloy - Electroless nickel plate per Specification MIL-C-26074, Class I.

3.6.1.5 Corrosion Resistant Primer Application. All mechanically prepared metal surfaces shall be primed with a corrosion inhibiting epoxy primer. Other metal surfaces, which require greater corrosion protection, shall be primed when required by the applicable drawing. The primer shall be applied according to manufacturer's recommendations. Bostik 463-6-3 primer, X-306 catalyst, and TL-52 thinner is an acceptable system. This primer shall dry at room temperature for 16 hours and shall then be cured in a clean, forced air oven for 1 hour at 150 °F plus 24 hours at 200 °F. Any other primer system must be tested and must meet all the qualifications of this specification.

3.6.1.6 Postcleaning Requirements. Precautions shall be taken to minimize contamination of the cleaned surfaces prior to application of the temperature control paint. Cleaned parts may be covered in a protective wrapping of clean, neutral, unplasticized film material such as Tedlar or equivalent material. Parts shall be painted as soon as possible after final cleaning or after being removed from the clean protective wrapping.

CAUTION: All operations subsequent to cleaning shall be performed by personnel wearing clean cotton, Dacron or nylon gloves, except as noted. Handling of parts and assemblies shall be kept to a minimum. Gloves shall be changed with sufficient frequency to assure cleanliness.

3.6.2 Masking. Surfaces that are to be free of paint shall be masked with pressure sensitive tape. Masked surfaces may be on unpainted metal, unpainted plastic, or painted parts. Parts that are subject to damage by tape removal such as thin gage materials, thin plating, or surfaces subject to possible contamination by tape adhesive, i.e., dry film lubricants, optical components, or other temperature control surfaces shall be so noted. Contact with temperature control surfaces shall be kept to an absolute minimum, and the operator's hands shall be clean, since handling of the parts with clean white

gloves may not be feasible during masking operations. Parts and assemblies containing holes or fasteners shall be suitably masked during surface preparation, coating, and stripping.

3.6.2.1 Masking Materials. Paper backed pressure sensitive adhesive tape shall be used for the majority of the surfaces to be masked. A cellophane or Mylar pressure sensitive tape of 1/2-mil nominal thickness may be used in special areas where a heavy build-up along the tape edge is undesirable.

3.6.2.2 Masking Procedure. Masking tape shall be applied in a manner that will seal all surfaces to be left unpainted or protected from paint overspray. Where contours exist, pulling of tape shall be avoided. Pressure shall be applied to seal progressively away from one end of the strip.

3.6.3 Application. The temperature control paint shall be applied to the surfaces to be coated in accordance with the requirements of the following paragraphs.

3.6.3.1 Equipment. The following equipment (or approved equal) shall be used for the application of primer and temperature control paint:

- (a) Spray booth, dry baffle or paint arrestor
- (b) Spray gun with suitable aircaps, fluid nozzles and paint cups.
- (c) Strainer
- (d) Viscosimeter, No. 2 Zahn cup or No. 4 Ford cup
- (e) Electronic paint thickness tester
- (f) Nylon gloves or expendable cotton gloves
- (g) Five micron air filter.

3.6.3.2 Primer. All bare metal surfaces prepared for painting shall be primed with General Electric SS-4044, and organic substrates with Union Carbide A-1100, before application of the temperature control paint. The primer shall be applied with an airbrush or spray gun with suction feed to a thickness of 0.05 to 0.20 mil. The primed surface shall dry at room temperature for not less than 1 hour nor more than 24 hours before applying the paint.

3.6.3.3 Preparation of Paint. The paint shall be prepared for spraying in accordance with the following procedure.

3.6.3.3.1 Mixing. The paint shall be mixed thoroughly prior to removing the paint from the container, using a device such as the Red Devil paint shaker. (This is necessary due to the tendency of the paint to settle heavily during storage.) The addition of the supplied catalyst to the paint shall be as specified in the following paragraph.

3.6.3.3.2 Catalyzing. Following the thorough mixing specified in 3.6.3.3.1, the catalyst shall be added at the ratio of 0.7 parts by weight catalyst solution to 100 parts by weight of S-13G/LO-1 paint. The paint shall be stirred thoroughly after the catalyst addition to assure complete dispersion of the catalyst. The solution shall be added only to the amount of paint that can be applied in a 1-hour period. Allow the catalyzed paint (after adjustment of the viscosity as specified in 3.6.3.3.3) to set for 10 minutes prior to application to the primed surfaces.

3.6.3.3.3 Thinning to Spraying Consistency. After adding the catalytic solution, the paint shall be mixed and thinned to a spraying consistency using X-99 thinner. The viscosity shall be measured at room temperature with either a No. 2 Zahn cup (20 to 27 seconds) or a No. 4 Ford cup (17 to 23 seconds). The paint shall be strained before application to the substrate. In the event that X-99 thinner prepared by the supplier is not available, thinner shall be prepared using the formulation specified below.

<u>Thinner Designation</u>	<u>Constituent</u>	<u>Specification</u>	<u>Percent (By Volume)</u>
X-99	Toluene	TT-T-548	40
	Xylene	TT-X-916	20
	Isopropanol	TT-I-735	20
	N-Butanol	TT-B-846	15
	N-Butyl Acetate	TT-B-838	5

3.6.3.4 Pressure and Gas. One of the following pressurizing gases shall be used for paint spraying.

- (1) Nitrogen meeting the requirements of Specification BB-N-411  
Type I, Class I, Grade A
- (2) Nitrogen obtained from the boil-off  
of liquid nitrogen
- (3) "Missile grade" air or nitrogen.

The pressure at the nozzle of the spray gun shall be sufficient to apply a smooth coating (approximately 30 to 35 pounds per

square inch (psi)). A 5-micron air filter (see 6.1(f)) shall be inserted into the gas pressurizing line to remove particulate matter and moisture.

**3.6.3.5 Spray Painting Procedure.** The catalyzed, thinned, and strained paint shall be applied over properly prepared metal or plastic surfaces. The paint shall be applied with a Binks Model No. 18 spray gun with pressure cup, or equal equipment. The line pressure and spray gun controls shall be adjusted to provide a smooth coating. Pressure required to obtain a uniform coating shall be determined by the operator. The coating shall be sprayed, preferably in a vertical plane, in a uniform thickness for each coat, allowing 10 minutes minimum between each coat for solvent evaporation. The paint shall be applied to provide the nominal thickness specified. Coating to nominal thickness shall preferably be completed within 5 hours after the start of painting. If this coating cannot be accomplished in 5 hours after the start of painting, no further paint coatings shall be applied until the paint already applied has cured for 64 hours at room temperature.

**3.6.3.6 Brush Painting Procedure.** If surface geometry of the parts to be coated is so complex that spray painting will not yield a coating of uniform thickness, brush coats of paint shall be applied to those areas which would be lightly coated if sprayed only. The brush-coated areas shall be overcoated with sprayed paint during spray application of paint to the parts. The number of brush coats to be applied shall be such that the combination of brushed and sprayed paint results in a coating of nominal thickness as required in 3.4.3. The coating shall be allowed to dry for 30 minutes between brush coats and between the final brush coat and first spray coat. The brushed paint shall be applied with a clean, high quality, chisel-shaped camel's hair brush of an appropriate size.

**Note:** The viscosity of the paint thinned for spraying may be too low for proper brushing. Paint thinning for brush painting shall be at the discretion of the painter.

**3.6.4 Curing.** The S-13G/LO-1 coating shall be air dried at room temperature for not less than 64 hours prior to handling.

**3.6.5 Demasking and Thickness Verification.** After the application of paint and prior to demasking, the coating thickness shall be measured to verify that the thickness requirement of

3.4.3 has been met. Demasking shall be performed carefully to avoid the starting of pulling stresses in the coating. In the event masking tape adhesive remains on the untreated surface, the surface shall be cleaned carefully, avoiding contact or contamination of the painted surface with solvents. To remove adhesive residue from the tape on a bare, unpainted surface, wipe the surface carefully with a swab of cotton or dacron saturated with a suitable solvent, follow with a toluene wipe, and allow to dry.

3.6.6 Cleaning and Reapplication. Soiled, damaged or thin-coated surfaces may be cleaned and/or recoated. Soiled surfaces shall be cleaned with Freon 113, ethyl alcohol conforming to Specification O-E-760, Grade I, Class B, or detergent.

(a) The Freon or ethyl alcohol shall be applied to coating to wet it thoroughly, and the coating shall then be wiped dry with lint-free cloth, paper or foam wipes. If the soiling is severe, the coating shall be wet down again and scrubbed with a plastic (nylon or polypropylene) bristle brush. Small marks shall be removed by scrubbing vigorously with clean, cotton cloths. The coating shall then be wet down again and wiped dry. Any steps in the above procedure may be repeated as often as required to remove the soil and/or contamination.

(b) The detergent shall be applied as a dilute solution (2 ounces per gallon) in distilled water using a clean sponge or soft, lint-free cloth. Vigorous cleaning shall be used to ensure removal of the soil. The coating shall then be rinsed thoroughly (repeatedly) with distilled water to remove the detergent and blotted dry.

Note: Reapplication to soiled surfaces is not required if the above cleaning removes the contamination and the coating then passes all applicable quality control requirements.

3.6.6.1 Reapplication to Damaged Surfaces. Scraped, gouged or otherwise damaged surfaces shall be repaired according to the following procedures. No post-cure (other than the 64 hour air-dry time) shall be required on repair coating unless the repair amounts to more than 5 percent of the total surface area of the hardware item.

3.6.6.1.6 Minor Damage Areas. Damaged areas less than approximately 0.25 square inches shall be repaired by removing all loose coating, vigorously cleaning the damaged area and surrounding coating with ethyl alcohol conforming to Specification O-E-760, Grade I, Class B, and reapplying the coating with a small camel's hair brush. If more than one coat application is required to obtain adequate thickness, 30 minutes drying time between coats shall be allowed.

Note: Neither the corrosion resistant primer nor the SS-4044 primer should be reapplied to minor damage areas. No sanding, feathering, or removal of additional coating, other than that obviously loosened, need be attempted.

3.6.6.1.2 Major Damage Areas. Damage areas greater than approximately 1/4 square inch shall be repaired by removing the coating in the damage area as completely as possible by scraping, and the area cleaned by multiple wiping with toluene conforming to Specification TT-T-548. If the damage area has the corrosion resistant primer and if this primer coat is damaged, the temperature control materials engineer shall be consulted on the necessity to repair the corrosion resistant primer. If repair of the corrosion resistant primer is required, the area shall be lightly sanded to remove all coating and SS-4044 primer, cleaned with toluene, and reprimed by brush application of the corrosion resistant primer. The damage area shall be reprimed with SS-4044 primer by applying one brush coat of the SS-4044 primer diluted with toluene at a ratio of one part SS-4044 to 10 parts toluene. After proper drying of the primer per 3.6.3.2, the repair coating shall be applied either by brush or spray gun.

Note: Care shall be taken to confine the repriming to the damage area only. Primer not covered by coating will darken during vacuum/ultraviolet exposure.

3.6.7 Special Handling. Service personnel shall be made aware of existence of the following:

- (a) Delicate components, especially if they protrude or are easily accessible to paint spray; electronics and components, thin gage materials, or other conditions requiring special handling.
- (b) Surfaces or components particularly sensitive to contamination by paint dust or specific contaminants shall be noted. Masking tape, hands, tools, or contaminants shall not be permitted contact with the painted surfaces.

3.7 Workmanship. Adequate care shall be taken to minimize contamination of the coating and the flight hardware under all work conditions throughout the process. All details of workmanship shall conform to the best practice for high quality processing and painting.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for Inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the procuring activity. The procuring activity reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Inspection. Spacecraft flight hardware and parts processed in accordance with the requirements of this specification shall be inspected in accordance with the following paragraphs. The painting process shall be monitored to verify that the requirements specified herein are enforced.

4.2.1 Prepainting Inspection. Surfaces to be coated shall be inspected prior to painting for conformance to surface preparation requirements of 3.6.1 and for contamination by oil, grease, dirt, biorganics and other foreign materials.

4.2.2 Coating Inspection. Coated surfaces shall be visually inspected for freedom from discoloration or other gross contamination, cracks, peel and blisters. The coating shall be adherent, uniform in color, and generally smooth. Minor roughness associated with coating repairs, unavoidable paint runs or small inclusions in the paint shall not be cause for rejection of the coating.

4.2.3 Coating Thickness Inspection. The coating thickness shall be measured on the part or on the reference sample, as appropriate, and recorded on the work order form. Excessive coating thickness at coating repairs shall be acceptable. Thickness at runs and sags which does not exceed three times the nominal shall be acceptable. Where possible, measurements shall be made only with a clean, polymer sheet between the measuring probe and the coated surface. If coating thickness is measured by other than direct reading on the part, the inspection record shall clearly indicate the method used.

4.2.4 Production Sample Inspection. Production control samples prepared in accordance with 3.4.2.1 shall be inspected. Optical property test methods and limits of acceptability shall be in accordance with the requirements of this specification.

#### 5. PREPARATION FOR DELIVERY AND SHIPPING OF COATED HARDWARE

5.1 Packaging and Protective Material. Packaging material shall consist of clean, neutral paper and clean, neutral, unplasticized film. Film materials suitable for wrapping covered, painted surfaces are: polyethylene, Tedlar, Teflon FEP, and Mylar. Neutral paper conforming to Specification MIL-P-17667 is suitable for external wrapping. Tape shall be used for closure.

5.2 Preparation for Storage. Surfaces coated in accordance with this specification shall be wrapped with suitable film as specified in the preceeding paragraph. If further protection is necessary, neutral kraft paper shall then be wrapped over the plastic film and secured with tape.

Note: Coated parts should not be packaged in zippered plastic film bags nor in heat-sealed plastic film envelopes.

## 6. PAINT ORDERING DATA

Procurement documents shall specify the following:

- (a) Title, number, and date of this specification
- (b) Quantity of material required, including thinner if desired
- (c) Size containers required, e.g., pints, quarts, etc.
- (d) Catalyst concentration required - undiluted or 10 percent solution
- (e) Method of shipment.

## 7. PREPARATION FOR DELIVERY AND SHIPPING OF PAINT

7.1 Packaging. The paint shall be supplied in wide-mouth glass or metal containers suitable for shipping, handling, and storage. When filled the containers shall be sealed airtight and shall be free from any damage which could cause leakage. The catalyst shall be supplied in vials or small-mouth bottles in the concentration specified.

7.2 Marking. Each container of paint shall be marked with a lot or batch number, the lot number of the silicone resin used, data of manufacture, manufacturer's name, date of purchase, and reference to this specification.

7.3 Packing. The paint and catalyst shall be prepared for shipment in accordance with best commercial practices in the manner that will assure carrier acceptance and safe delivery and shall meet, as a minimum, the requirements of carrier rules and regulations applicable to the mode of transportation.

7.4 Shipping. The mode of transportation selected shall minimize the exposure of the paint and catalyst to high temperatures which may adversely affect shelf-life stability.

Note: The procuring agency shall be responsible to obtain expedited delivery of the paint and catalyst from the receiving point to the place of refrigerated storage.

8. CHANGES, DEVIATIONS, OR WAIVERS

No technical changes, deviations, or waivers will be made to the requirements of this document without the approval of the cognizant engineering activity of MSFC.

Custodian:

NASA-George C. Marshall Space  
Flight Center

Preparing Activity:

George C. Marshall Space  
Flight Center

**APPENDIX B**  
**TML/VCM TEST DATA SHEETS**



**Aerospace Systems Division**

P O Box 1062, Boulder, Colorado 80306-1062 (303) 939-4000 TWX 910-940-3241 Telex 45-605 Cable BAREC

TO: IITRI  
10 West 35th Street  
Chicago, IL 60616

Date: January 21, 1985  
Ref. P.O. 39798  
BASD 3150-284-00  
84260-85.037

Attn: Richard Mell

MATERIAL: One specimen of Silicone rubber, clear, P/N J-182A

RTV-602/LO

Prior to testing the sample was conditioned for  
24 hours at 25°C and 50% RH.

TEST: BASD 33074 - "Method: Short Term Weight Loss and  
Condensed Contaminant Testing of Materials in a  
Thermal Vacuum Environment"

The test consists of exposure of the specimen to  
a 125°C, 10<sup>-5</sup> Torr environment for 24 hours with  
a collector at 25°C for collection of the Volatile  
Condensable Materials (VCM).

**RESULTS:**

Test No.	Weight Loss, %	VCM %	Forward Light Scatter, %/cm <sup>2</sup>	Visible VCM Deposit?
85-5298	0.406	0.027	5.42 X 10 <sup>-1</sup>	Yes

Refer to details on attached data sheets.

  
K.G. Roller  
Principal Member Technical Staff

KGR/slh

Attachments

cc: J. Birkeness

VACUUM EXPOSURE NEPHELOMETER TEST - VEN NO. 5298

TVL NO. \_\_\_\_\_

DATE SUBMITTED 1-9-85TESTED BY L.C.DATE STARTED 1-15-85CHECKED BY KER

DATE REPORTED \_\_\_\_\_

MATERIAL clear  
TYPE Silicone RubberPART NO. J-182 ASPEC OR  
STD NO. \_\_\_\_\_MIT  
NO. \_\_\_\_\_MFG. IITRI

TRADE NAME/NO. \_\_\_\_\_

LOT NO. \_\_\_\_\_

MIX RATIO\* \_\_\_\_\_

CURE/POST CURE\* \_\_\_\_\_

VAC BAKE\* \_\_\_\_\_

OTHER INSTRUCTIONS TEST, HUMIDIFY PER BMSD 33074REQUESTOR Roller/Victor HUMPHREYSPHONE - 4209  
PROGRAM (312) 567-4362PG 39798  
CHG. NO. 3150-284-00VACUUM EXPOSURE\*: Chamber# 1 Final Pressure  $1.2 \times 10^{-6}$  TorrSample Temp. 125 °COptic Temp. 25 °CTotal Time at Test Temp. 24 hr. Sample-to-Optic Distance 9 cm

Test Temp. \_\_\_\_\_ Optical Quality \_\_\_\_\_ Misc. \_\_\_\_\_

SAMPLE: Size\*  $2.8 \times 3.5 \times 0.43$  cm Surface Area 25 cm<sup>2</sup>

Substrate Weight \_\_\_\_\_ g Substrate Material \_\_\_\_\_

Sample &amp; Subs. Wt: Before \_\_\_\_\_ g After \_\_\_\_\_ g

Sample Wt. Before 4.05064 g After 4.03421 gWt. Loss 0.01643 g Wt. Loss 0.406 % Wt. Loss  $6.57 \times 10^{-4}$  g/cm<sup>2</sup>

## CONDENSED OUTGASSED PRODUCTS (COP):

Optic Weight: Clean 1.94385 g Contaminated 1.94493 gWt. of Contaminant 0.00108 g COP 0.037 % COP  $4.32 \times 10^{-5}$  g/cm<sup>2</sup>Visible Contaminant Film YES ☒ NO ☐ Sensitivity \_\_\_\_\_NEPHELOMETER: Film Exposure Time 1/25 sec Incident Light 295 mv

Readings

After

Before

Optic 40.00 mv 0.060 mvBackground 0.060 mv 0.060 mv(39.94) - (0.000) = 39.94 net mvnet mv/cm<sup>2</sup> 1.598 % scatter/cm<sup>2</sup>  $5.42 \times 10^{-1}$  Sensitivity \_\_\_\_\_

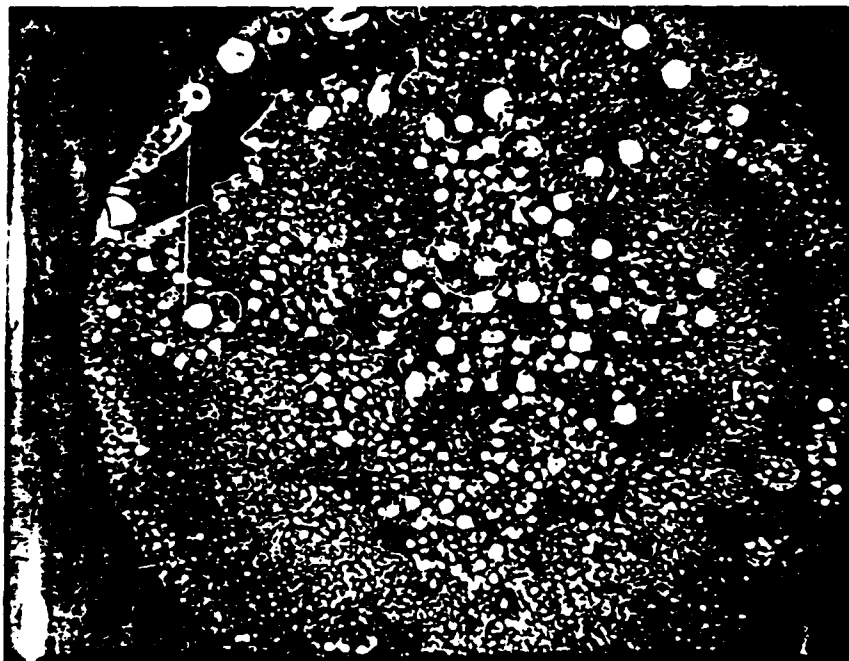
VEN CATEGORY(S) \_\_\_\_\_

NO. \_\_\_\_\_

Data Validity \_\_\_\_\_

Enter subcontractor data on other side

REMARKS: \*Some material stuck to Petri Dish after initial weighing☒ Sample thickness is greater than 0.3 cm



5298 40.0 - 0.060



**Aerospace Systems Division**

P.O. Box 1062, Boulder, Colorado 80306-1062 (303) 939-4000 TWX 910-940-3241 Telex 45-605 Cable BAREC

TO: IITRI  
10 West 35th Street  
Chicago, IL 60616

Date: January 21, 1985  
Ref. P.O. 39798  
BASD 3150-284-00  
84260-85.036

Attn: Richard Mell

MATERIAL: One specimen of Silicone rubber, light blue, P/N J-182C RTU-12

Prior to testing the sample was conditioned for  
24 hours at 25°C and 50% RH.

TEST: BASD 33074 - "Method: Short Term Weight Loss and  
Condensed Contaminant Testing of Materials in a  
Thermal Vacuum Environment"

The test consists of exposure of the specimen to  
a 125°C,  $10^{-5}$  Torr environment for 24 hours with  
a collector at 25°C for collection of the Volatile  
Condensable Materials (VCM).

**RESULTS:**

Test No.	Weight Loss, %	VCM %	Forward Light Scatter, %/cm <sup>2</sup>	Volatile VCM Deposit?
85-5297	0.243	0.034	$6.82 \times 10^{-3}$	Yes

Refer to details on attached data sheets.

*K.G. Roller*  
K.G. Roller

Principal Member Technical Staff

KGR/slh

Attachments

cc: J. Birkeness

VACUUM EXPOSURE NEPHELOMETER TEST - VEN NO. 5297

TVL NO. \_\_\_\_\_

DATE SUBMITTED 1-9-85TESTED BY L.C.DATE STARTED 1-15-85CHECKED BY KAR

DATE REPORTED \_\_\_\_\_

MATERIAL LT Blue  
TYPE Silicone RubberPART RTV-12  
NO. J-182CSPEC OR  
STD NO. \_\_\_\_\_ MIT  
NO. \_\_\_\_\_MFR. IITRI

TRADE NAME/NO. \_\_\_\_\_ LOT NO. \_\_\_\_\_

MIX RATIO\* \_\_\_\_\_ CURE/POST CURE\* \_\_\_\_\_ VAC BAKE\* \_\_\_\_\_

OTHER INSTRUCTIONS HUMIDIFY, TEST PER ASD 33074REQUESTOR Roller/VICTOR HUMPHREYS PHONE 4209 P.O. 39798  
PROGRAM (312) 667-4632 CHG. NO. 3150-284-00VACUUM EXPOSURE:\* Chamber# 3 Final Pressure  $7.0 \times 10^{-7}$  TorrSample Temp. 125 °C Optic Temp. 25 °CTotal Time at Test Temp. 24 hr. Sample-to-Optic Distance 9 cm

Test Temp. \_\_\_\_\_ Optical Quality \_\_\_\_\_ Misc. \_\_\_\_\_

SAMPLE: Size\*  $3.0 \times 3.3 \times 0.41$  cm Surface Area 25 cm<sup>2</sup>

Substrate Weight \_\_\_\_\_ g Substrate Material \_\_\_\_\_

Sample &amp; Subs. Wt: Before \_\_\_\_\_ g After \_\_\_\_\_ g

Sample Wt. Before 3.98652 g After 3.97684 \*Wt. Loss 0.00968 Wt. Loss 0.243% Wt. Loss  $3.87 \times 10^{-4}$  g/cm<sup>2</sup>

## CONDENSED OUTGASSED PRODUCTS (COP):

Optic Weight: Clean 1.94385 g Contaminated 1.94520 gWt. of Contaminant 0.00135 g COP 0.034% COP  $5.40 \times 10^{-5}$  g/cm<sup>2</sup>Visible Contaminant Film YES ☒ \*\* NO ☐ Sensitivity \_\_\_\_\_NEPHELOMETER: Film Exposure Time 1/25 sec Incident Light 295 mv

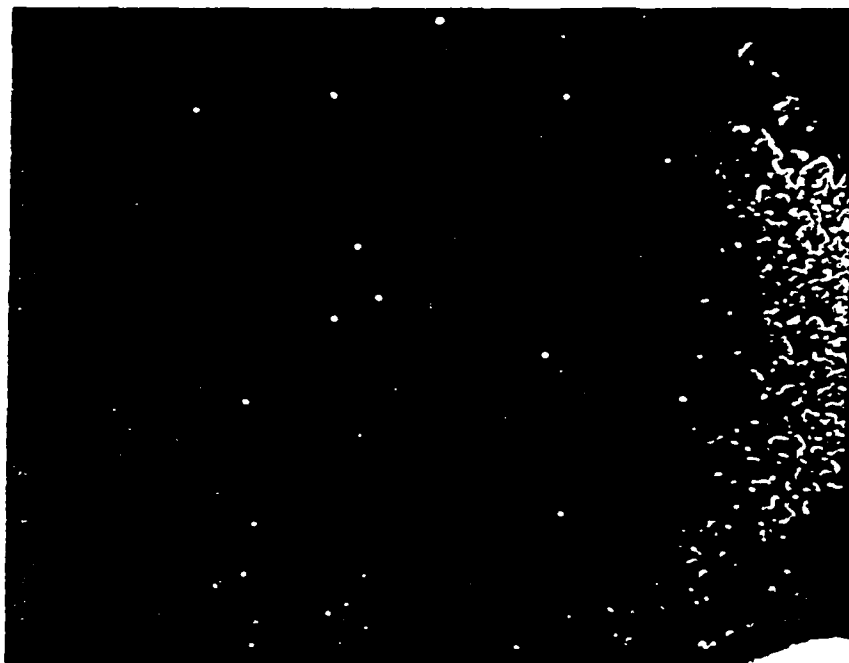
Readings After Before

Optic 0.563 mv 0.060 mvBackground 0.060 mv 0.060 mv(0.503) - (0.000) = 0.503 net mvnet mv/cm<sup>2</sup>  $2.01 \times 10^{-2}$  % scatter/cm<sup>2</sup>  $6.82 \times 10^{-3}$  Sensitivity \_\_\_\_\_

VEN CATEGORY(S) \_\_\_\_\_ NO. \_\_\_\_\_ Data Validity \_\_\_\_\_

Enter subcontractor data on other side

REMARKS: \* Some material stuck to ven chamber screen and thermocou\*\* Sample lost Blue tint, ven Disk gained a blue tint☒ Sample thickness is greater than 0.3 cm.



5297 0.563-0.060



TEST REPORT FOR TOTAL MASS LOSS AND  
COLLECTED VOLATILE CONDENSIBLE MATERIALS

TESTING DONE IN ACCORDANCE WITH ASTM E-595-77  
AND NASA SP-R-0022A SPECIFICATIONS

TESTED FOR: IIT Research Institute

10 W. 35th Street

Chicago, IL 60616

ATTN: Richard Mell

PRODUCT TESTED: Silicone Film RTV-62

LOT NUMBER: J182D

DESCRIBE HERE ALL PROCESSING DONE BY McGHAN NuSIL PERSONNEL, INCLUDE  
RECIPE, CURE TIME AND TEMPERATURE, AND ANY OTHER SPECIFIC CONDITIONS.

tested as received

TEST NUMBER: 295

HUMIDIFICATION CYCLE: 24 HOURS 22 °C TEMP. 50 % R.H.

TEST DATE: 1/16/85 PERIOD: 24 HRS.

PRESSURE:  $7.5 \times 10^{-6}$  TORR, SPECIMEN TEMP: 125 °C COLLECTOR TEMP: 25 °C

RESULTS:

TOTAL MASS LOSS (TML): 0.12%

COLLECTED VOLATILE CONDENSIBLE MATERIAL (CVCM): 0.01%

WATER VAPOR RECOVERED (WVR): 0.01%

OBSERVATIONS:

CVCM APPEARANCE ON COLLECTOR PLATES:

☒ THIN, ☐ HEAVY, ☐ OPAQUE, ☐ TRANSPARENT, Blue COLOR,

☒ INTERFERENCE FRINGES, ☐ SOLID, ☐ LIQUID, ☐ EXCESS

LIQUID RUNS, DEPOSIT COVERS 60 - 70 % OF COLLECTOR DISC.

OTHER COMMENTS:

SPECIMEN APPEARANCE AFTER TEST: No change

BY: Brett A. Ray  
Quality Assurance Manager

P.O. # 39799

DATE: Januray 18, 1985



TEST REPORT FOR TOTAL MASS LOSS AND  
COLLECTED VOLATILE CONDENSIBLE MATERIALS  
TESTING DONE IN ACCORDANCE WITH ASTM E 595-77  
AND NASA SP-R-0022A SPECIFICATIONS

TESTED FOR: IIT Research Institute  
10 West 35th Street  
Chicago, IL 60616  
ATTN: Richard Mell

PRODUCT TESTED: Silicone Film RTV 602/40  
LOT NUMBER: J182B

DESCRIBE HERE ALL PROCESSING DONE BY McGHAN NuSIL PERSONNEL, INCLUDE  
RECIPE, CURE TIME AND TEMPERATURE, AND ANY OTHER SPECIFIC CONDITIONS.  
tested as received

TEST NUMBER: 295  
HUMIDIFICATION CYCLE: 24 HOURS 22 °C TEMP. 50 % R.H.  
TEST DATE: 1/16/85 PERIOD: 24 HRS.  
PRESSURE:  $7.5 \times 10^{-6}$  TORR, SPECIMEN TEMP: 125 °C COLLECTOR TEMP: 25 °C

RESULTS:

TOTAL MASS LOSS (TML): 0.28 %  
COLLECTED VOLATILE CONDENSIBLE MATERIAL (CVCM): 0.00 %  
WATER VAPOR RECOVERED (WVR): 0.01 %

OBSERVATIONS:

CVCM APPEARANCE ON COLLECTOR PLATES:

X THIN, \_\_\_\_\_ HEAVY, \_\_\_\_\_ OPAQUE, \_\_\_\_\_ TRANSPARENT, Blue COLOR,  
X INTERFERENCE FRINGES, \_\_\_\_\_ SOLID, \_\_\_\_\_ LIQUID, \_\_\_\_\_ EXCESS  
LIQUID RUNS, DEPOSIT COVERS 50 - 60 % OF COLLECTOR DISC.

OTHER COMMENTS:

SPECIMEN APPEARANCE AFTER TEST: No change

BY:   
Quality Assurance Manager

P.O. # 39799

DATE: 1/18/85

**APPENDIX C**  
**SCEPTRE TEST RUN 85-QV-01**



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*The University of Dayton*

Test Report:

SCEPTRE test run 85-QY-01

Synergistic testing of S-13G/LD

John. D. Ruley

August 1985

C-02

PRELIMINARY REPORT  
SCEPTRE TEST RUN 85-QV-01

ABSTRACT

In support of the S-13G/LO requalification effort, two dimethyl silicone binders (GE RTV-12 and SWS V-10) were evaluated for space stability under vacuum/ultraviolet/charged particle conditions as components of a white thermal control coating (S-13G/LO). For comparison purposes, specimens of the original S-13G/LO coating (GE RTV-602 silicone binder) were tested. After 700 ESH of exposure, the RTV-12 based coating had a solar absorptance increase of +.039; while the V-10 based coating had increased by +.025. The RTV-602 based original coating had an increase of +.024 after 350 ESH.

INTRODUCTION

SCEPTRE (Space Combined Effects Primary Test Research Equipment) is the Air Force Materials Laboratory's primary device for performance testing of spacecraft thermal control materials. It is capable of simulating the synergistic effects of vacuum, ultraviolet radiation, and electron radiation. For this test a vacuum level in the  $10^{-7}$  torr range was used, with a UV flux of 1.0 to 2.5 EUVS (depending on the sample position) and an electron flux of  $6 \times 10^9$  to  $12 \times 10^9$  e-/cm<sup>2</sup>-sec (again, depending on the sample position).

In Test Run 85-QV-01, two candidate materials provided by the IIT Research Institute (IITRI) for the Air Force's S-13G/LO replacement program were tested for 700 equivalent sun hours. S-13G/LO is a white thermal control coating, consisting of a potassium silicate encapsulated zinc oxide pigment in a dimethyl silicone (RTV-602) binder. General Electric has discontinued production of the RTV-602 silicone, and the IITRI is under contract to develop a coating of comparable performance using another silicone.

The two candidate materials tested incorporated the same potassium silicate encapsulated zinc oxide pigment used in the original S-13G/LO formulation, but the binder materials were different. One sample used GE RTV-12, while the other used SWS V-10. Like RTV-602, both of these materials are dimethyl silicones.

One specimen of each candidate material was tested in SCEPTRE. For comparison purposes, two specimens of the original S-13G/LO formulation were tested, along with one anodized aluminum specimen (Alcoa Corporation Alzak, 0.1 mil thickness); all of which were exposed to the ionizing radiation environment. A second Alzak specimen was used as a control, along with a black velvet paint specimen and two additional S-13G/LO's. These specimens were not exposed to ionizing radiation.

#### TEST CONDITIONS

In SCEPTRE, exposure conditions vary with position on the sample wheel. The exposure conditions are shown in Table 1.

With the exception of the low-energy electron flux, these conditions were maintained throughout the run, except when the exposure was stopped for measurements.

#### TEST RESULTS

Absorptance data is summarized in Table 2. This data presents a few anomalies, primarily in regard to pre- and post-test measurements in air. It should be noted that the DK-2 data is inherently more accurate, due to its larger integrating sphere and more efficient optical system.

Figures 1 through 5 represent the results of the spectrophotometer experiment expressed in terms of absolute reflectance as a function of wavelength for the five samples exposed to radiation. The data is plotted for the pre-test air and vacuum; and post-test air and vacuum measurement points, with equivalent ESH values on the plots.

In-situ absorptance data is plotted in Figures 6 through 10 for the five samples exposed to ionizing radiation. The symbols on these plots are the actual averaged absorptances at each test point, including an estimated point from the 24 hour data, which was taken manually. The solid curve is from the equation:

$$A_s = A_0 + A_1(1 - e^{-t/T_0})$$

where

$A_s$  = sample absorptance

$A_0$  = initial absorptance

$t$  = exposure time (hours)

$A_1$  = maximum change in solar absorptance

$T_0$  = time constant

where  $A_0$  and  $A_1$  have been determined by a least-squares fit technique, and  $T_0$  by iteration.

A comparison of Figures 8 and 9 illustrates the superiority of V-10 to RTV-12. The V-10 appears to curve over and approach an asymptote of  $\alpha_s \sim .24$ , while the RTV-12 continues to degrade in a linear fashion. Indeed, comparison of Figures 9, 10, and 11 suggests that the RTV-12 is quite similar to the original material (RTV-602).

Figure 11 is a plot of differential reflectance (initial reflectance minus terminal reflectance) for RTV-12, V-10, and S-13G/LO. Note that the data is scattered through a 2% range, as expected (the spectrophotometer is accurate to  $\pm 1\%$ ). Again, the superiority of V-10 is obvious. The 425 nm absorption peak for V-10 is only about as high as that for the S-13G/LO, even though this specimen has been exposed at twice the UV intensity.

Figure 12 shows the same information for the Alzak specimens. The oscillation is due to interference fringe shifting in the anodized surface, and does not represent noise.

Figures 1 through 4 illustrate a considerable recovery behavior for the materials. This effect is also further illustrated by Figure 13. To produce Figure 13, the RTV-12 based specimen was rotated into measurement position at the end of the

test and the spectrophotometer was set to time drive mode at 425 nm. The air valve was opened at the four minute mark. A virtually instantaneous 2% reflectance increase was noted, which increased by another 1% in the next 24 hours. This result tends to validate the arguments against ex-situ measurement of such materials.

#### CONCLUSION

Test Run 85-QV-01 provided sufficient data to show the clear superiority of SWS V-10 silicone over both RTV-12 and RTV-602. In addition, this test run demonstrated SCEPTRE's new capability for:

1. Multiple sun exposure (2.5 EUVS maximum in this configuration)
2. Multiple intensities in a single test run (1, 2, 2.5 EUVS)
3. Electron exposure
4. Measurement of electron flux levels
5. Measurement of in-situ reflectances
6. Identification of recovery phenomena.

In future operations, it is expected that these capabilities will be fully exploited.

TABLE 1  
EXPOSURE CONDITIONS

<u>Sample</u>	<u>Sun Level (EUVS)</u>	<u>Low-Energy<sup>1</sup> e-flux (400 eV) (e-/cm<sup>2</sup>-sec)</u>	<u>High-Energy e-flux (8 KeV) (e-/cm<sup>2</sup>-sec)</u>
S-13 #1	1.0	$6 \times 10^{10}$	$6 \times 10^9$
S-13 #2	1.0	$6 \times 10^{10}$	$6 \times 10^9$
RTV-12	2.0	$9 \times 10^{10}$	$9 \times 10^9$
V-10	2.0	$9 \times 10^{10}$	$9 \times 10^9$
Alzak #12	2.5	$12 \times 10^{10}$	$12 \times 10^9$
Alzak #9	-----	unexposed	-----
Black Velvet	-----	unexposed	-----
S-13 #3	-----	unexposed	-----
S-13 #4	-----	unexposed	-----

<sup>1</sup> Low energy gun was only operated for 24 hours

TABLE 2

## SOLAR ABSORPTANCES

Sun Level (EUVS)	air (DK-2)	air (M-IV)	0		24.000		160.000		350.000		air		change
			hrs *	in-situ	hrs *	in-situ	hrs *	in-situ	hrs *	in-situ	post	post	
sample:													
S-13 # 1	1.000	0.211	0.195	0.205	0.210	0.212	0.230	0.215	0.232	0.025			
S-13 # 2	1.000	0.211	0.196	0.205	0.210	0.212	0.227	0.214	0.236	0.022			
RTV-12	2.000	0.231	0.207	0.216	0.221	0.235	0.255	0.240	0.258	0.039			
V-10	2.000	0.224	0.203	0.208	0.213	0.225	0.233	0.220	0.239	0.025			
AZ # 12	2.500	0.277	0.221	0.247	0.292	0.325	0.327	0.284	0.325	0.080			
AZ # 9	0	0.258	0.207	0.221	0.221	0.220	0.221	0.223	0.273	0			
B.V	0	0.980	0.980	0.981	0.981	0.981	0.981	0.981	0.981	0			
S-13 # 3	0	0.215	0.197	0.205	0.206	0.206	0.209	0.209	0.226	0.004			
S-13 # 4	0	0.214	0.197	0.213	0.212	0.210	0.213	0.209	0.227	0			

\* - Note: to get ESH, multiply hrs by Sun Level

## Legend:

S-13: G.E. RTV-602 silicone based coating  
 RTV-12: G.E. RTV-12 silicone based coating  
 V-10: S.W.S. V-10 silicone based coating  
 B.V.: 3M Corp. 'Black Velvet' paint  
 AZ: Alcoa Corp. 'Alzak' .1 mil anodized aluminum

S-13 # 1

— : pre - test (air)  
- - - : pre - test (vac)  
..... : post - test (vac)  
..... : post - test (air)

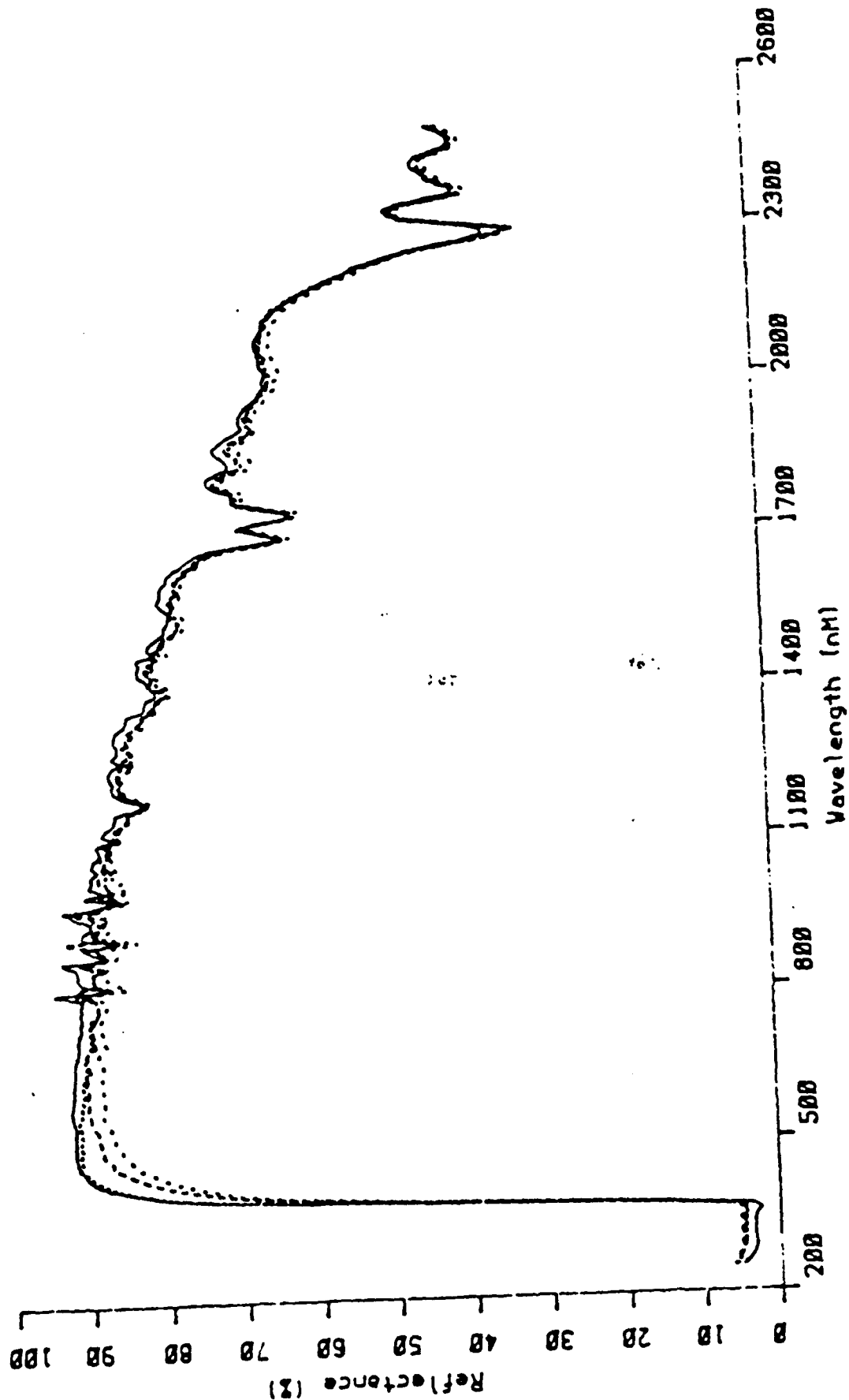


Figure 1

S-13 • 2

! pre - test (air)  
! pre - test (vac)  
! post - test (vac)  
! post - test (air)

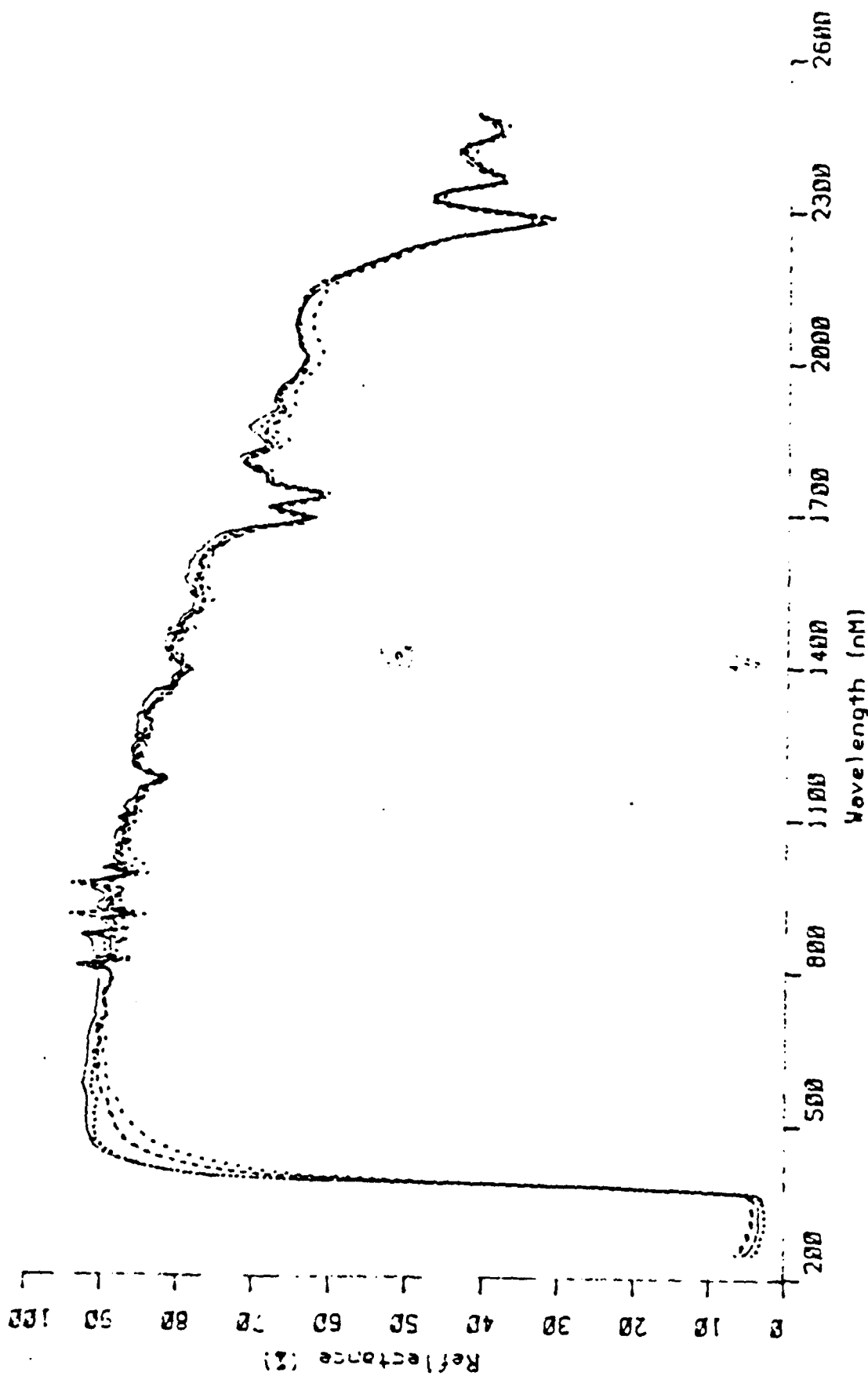


Figure 2

RTV-12

..... : pre - test (air)  
..... : pre - test (vac)  
..... : post - test (vac)  
..... : post - test (air)

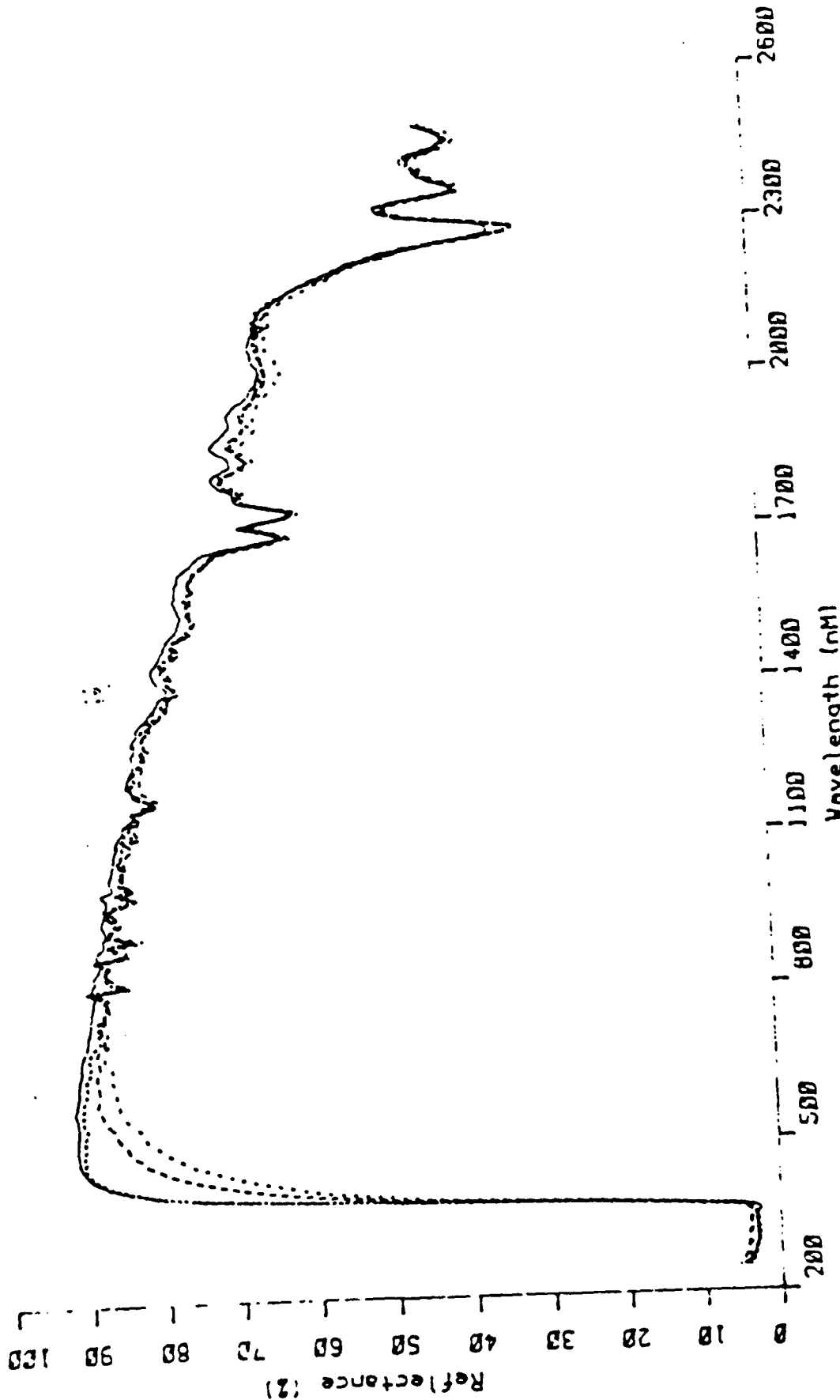


Figure 3

V-10

- : pre - test (air)  
 - : pre - test (vac)  
 - : post - test (vac)  
 - : post - test (air)

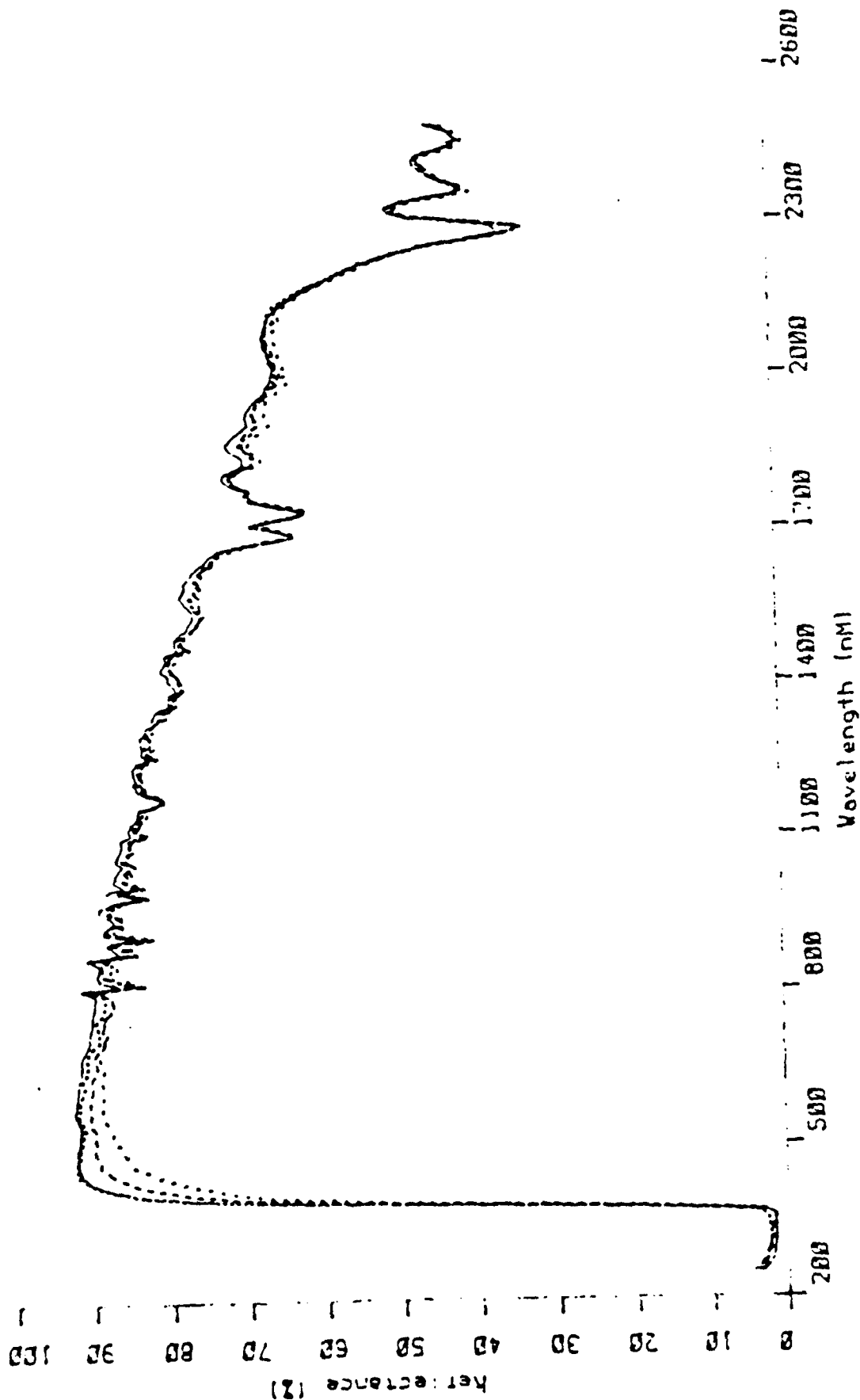


Figure 4

Alzak # 12

. : pre - test (air)  
 ..... : pre - test (vac)  
 ..... : post - test (vac)  
 - - - - : post - test (air)

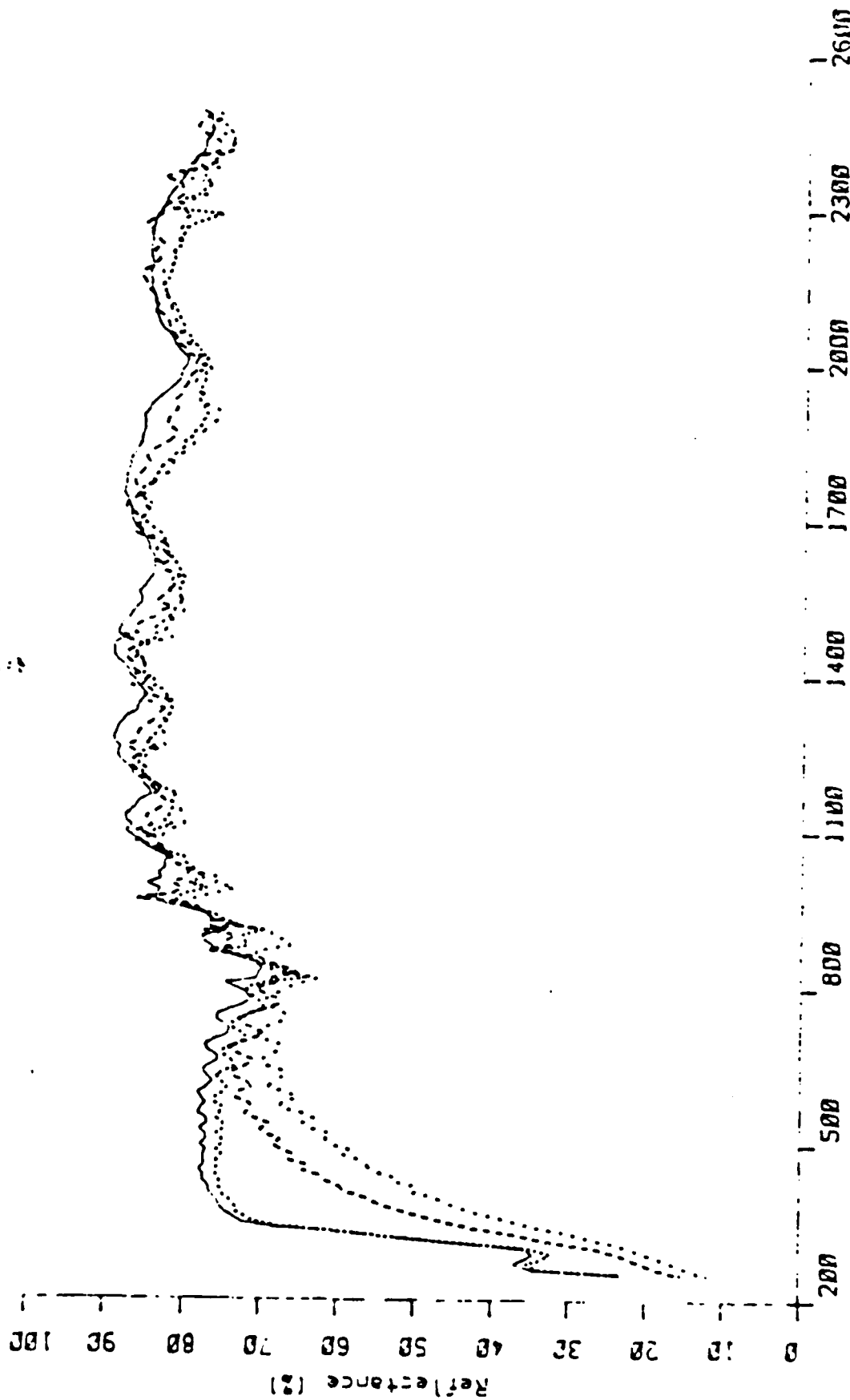
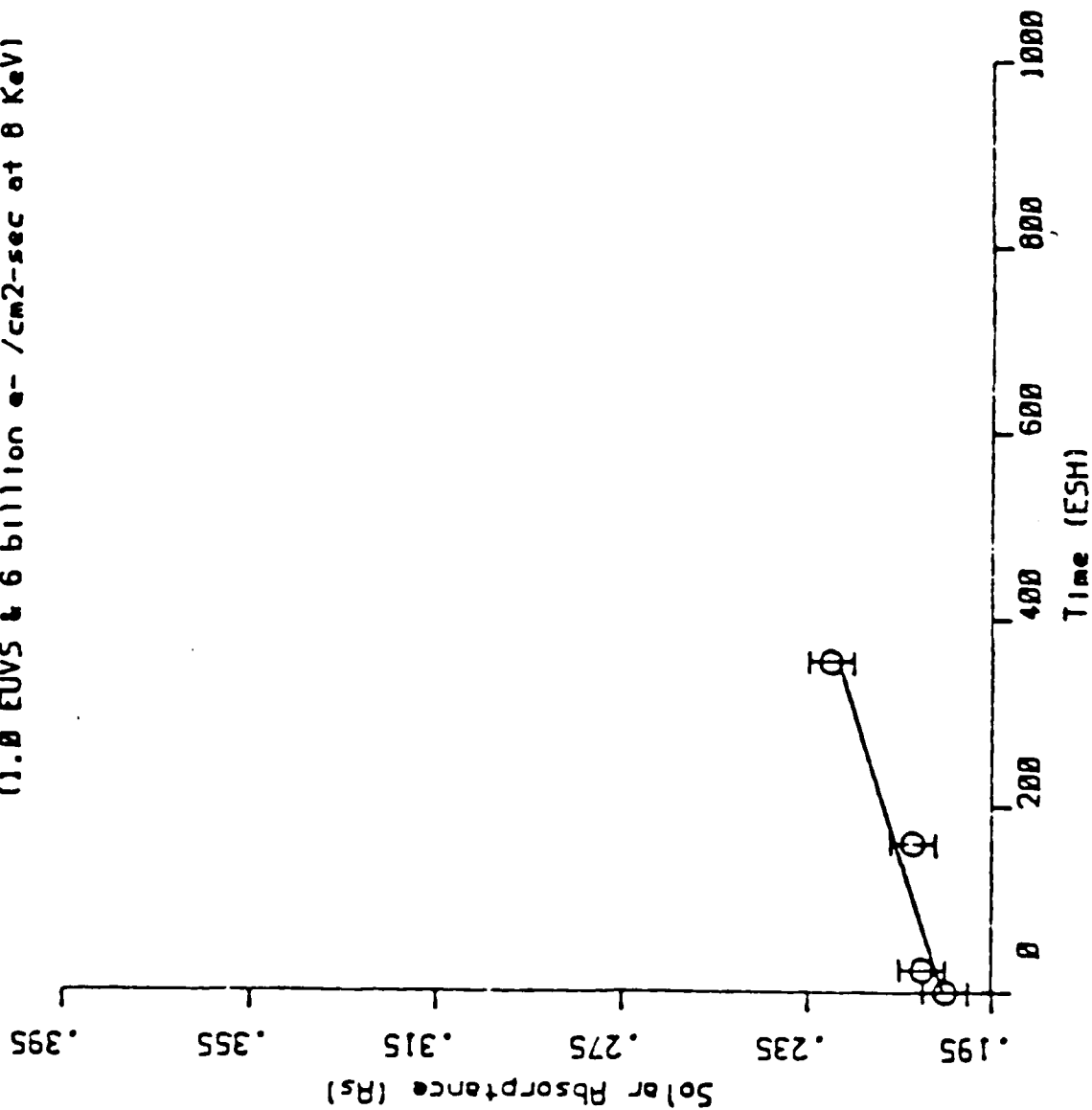


Figure 5

S-13 a 1

(1.0 EUVS & 6 billion e- /cm2-sec at 0 KeV)



○ : Measured

— : Calculated from iterative fit to exponential equation

Date Points:  
time As

0.0	.205
24.0	.210
160.0	.212
353.0	.230

Figure 6

S-13 # 2

(1.0 EUVS & 6 billion e- / cm<sup>2</sup>-sec at 0 KeV)

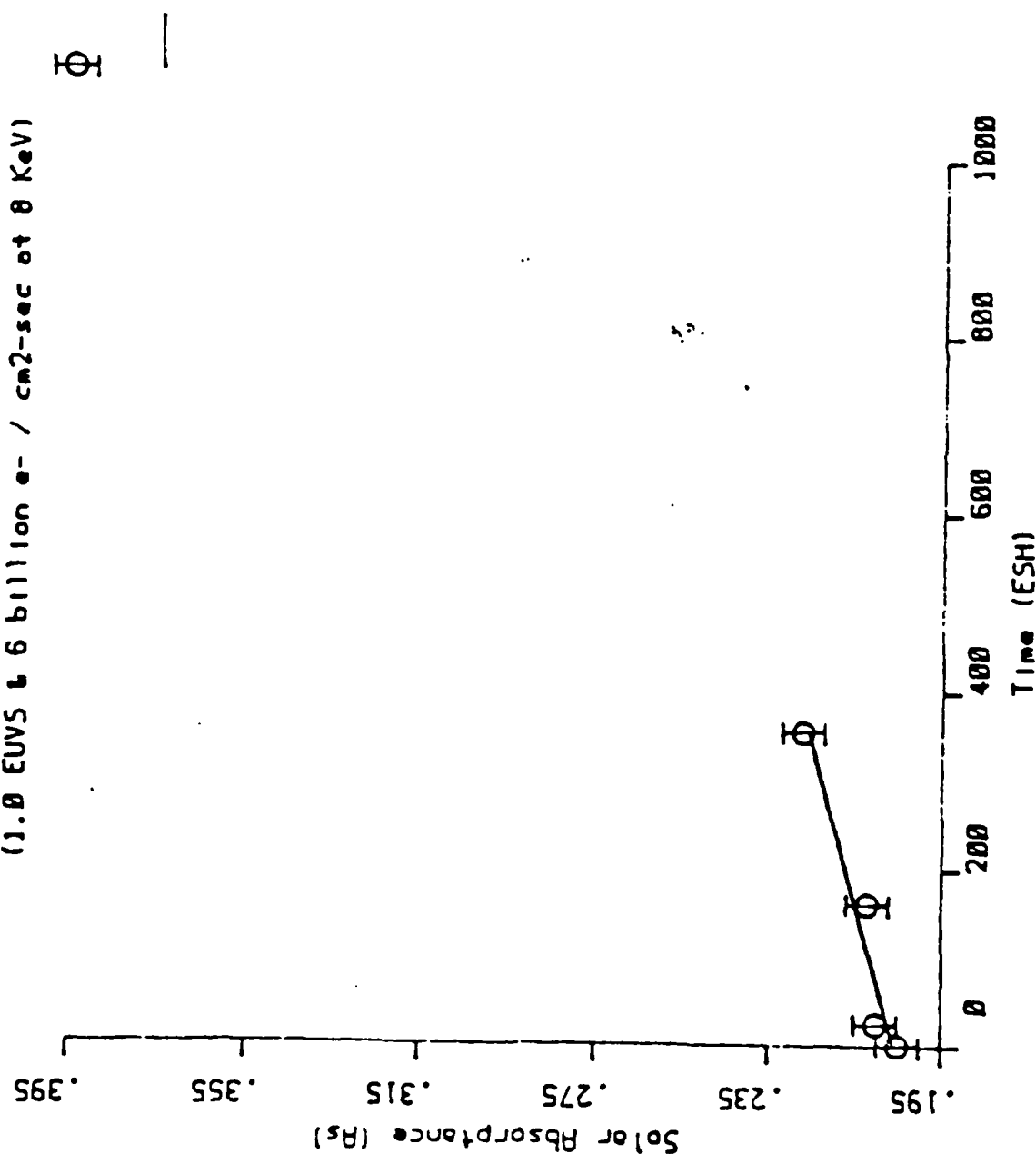


Figure 7

RTV - 12

(2.0 EUVS & 9 billion e- / cm<sup>2</sup>-sec at 0 KeV)

$\Phi$

: Measured

--- : Calculated from  
iterative fit to  
exponential equation

Date Points:  
time  $R_s$

0.0	.216
48.0	.221
320.0	.235
706.0	.255

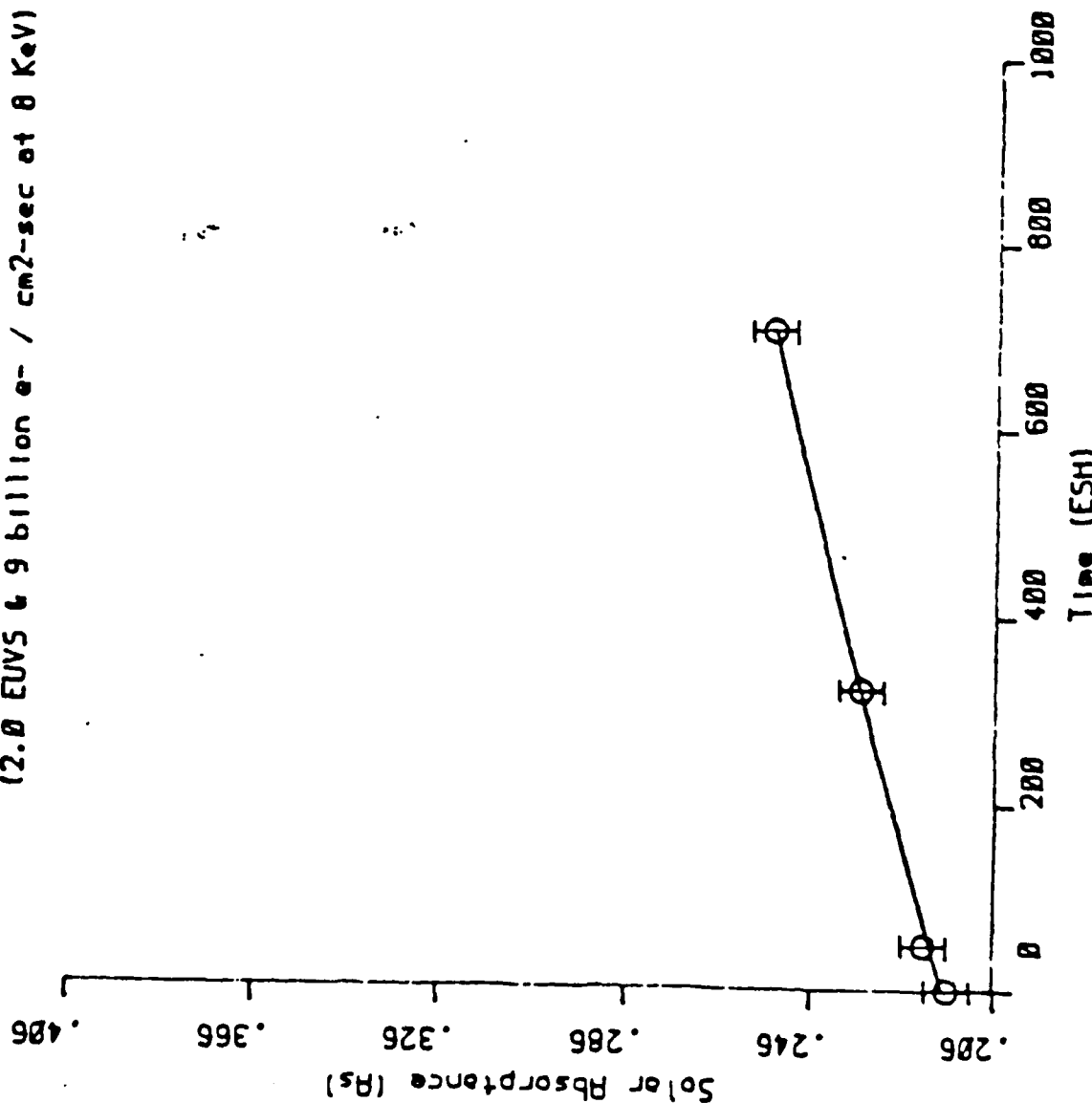
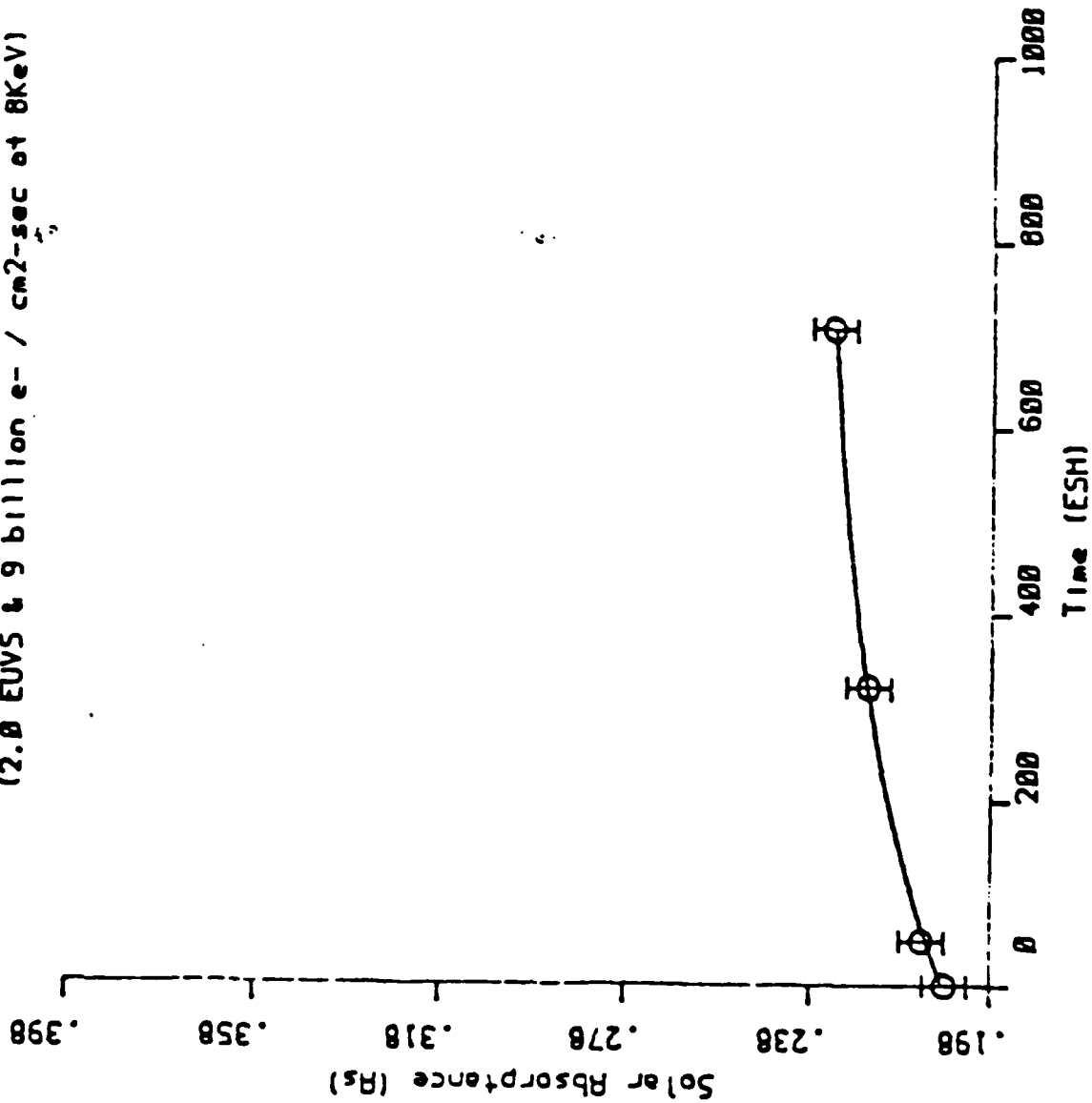


Figure 8

V - 1B

(2.8 EUVS & 9 billion e- / cm<sup>2</sup>-sec at 8KeV)



Φ : Measured

--- : Calculated from  
iterative fit to  
exponential equation

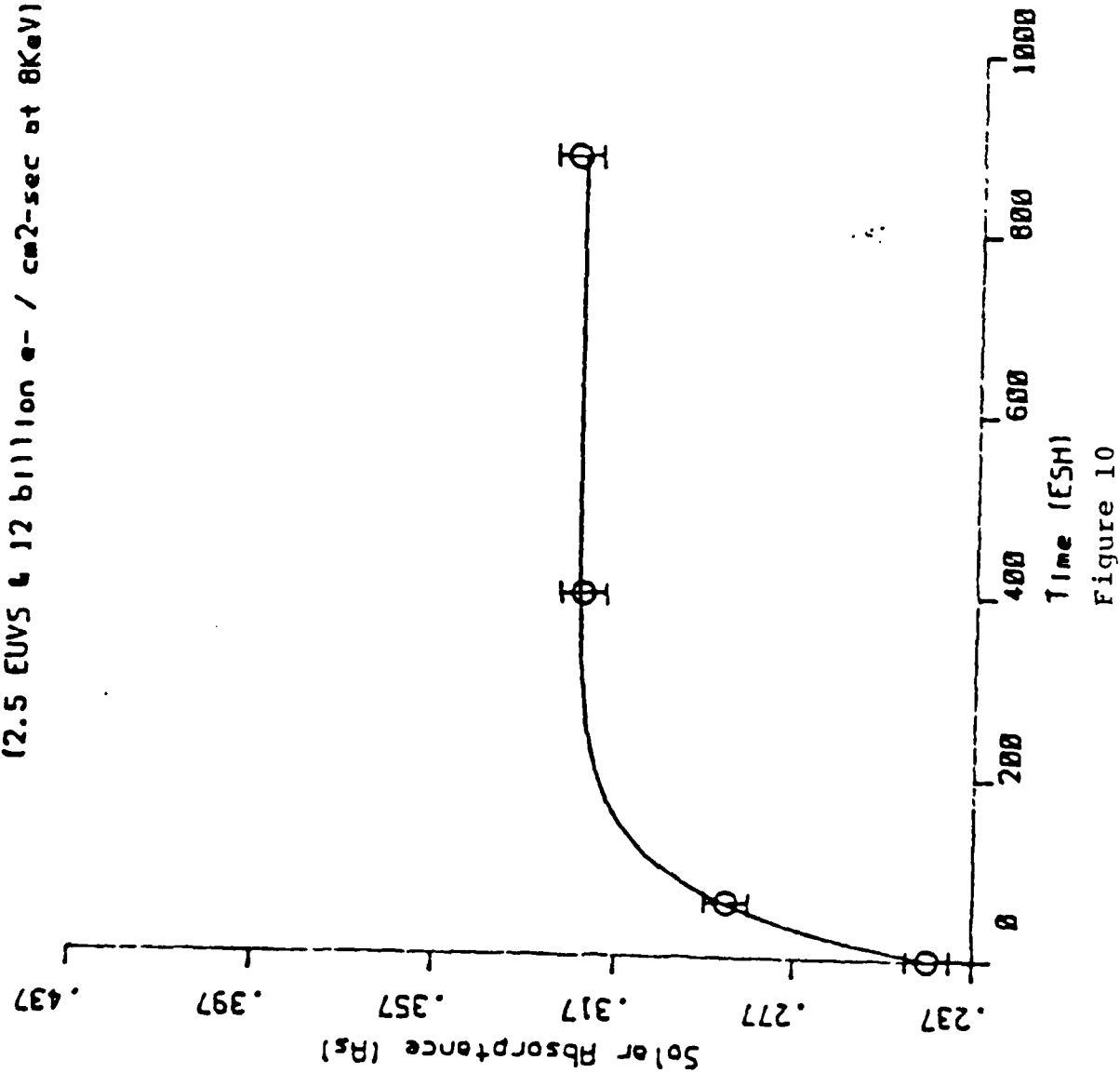
Data Points:  
time As

0.0	.208
48.0	.213
320.0	.225
706.0	.233

Figure 9

Alzak # 12

(2.5 EUVS & 12 billion e- / cm<sup>2</sup>-sec at 8KeV)



$\Phi$  : Measured

— : Calculated from  
iterative fit to  
exponential equation

Data Points:  
time  $R_s$

0.0	.247
60.0	.292
400.0	.325
883.0	.327

Figure 10

S-13 Candidate Material Performance

-	:	S-13	• 1	(1.0 EUVS
.....	:	S-13	• 2	(1.0 EUVS
.....	:	RTV-12		(2.0 EUVS
.....	:	V-10		(2.0 EUVS

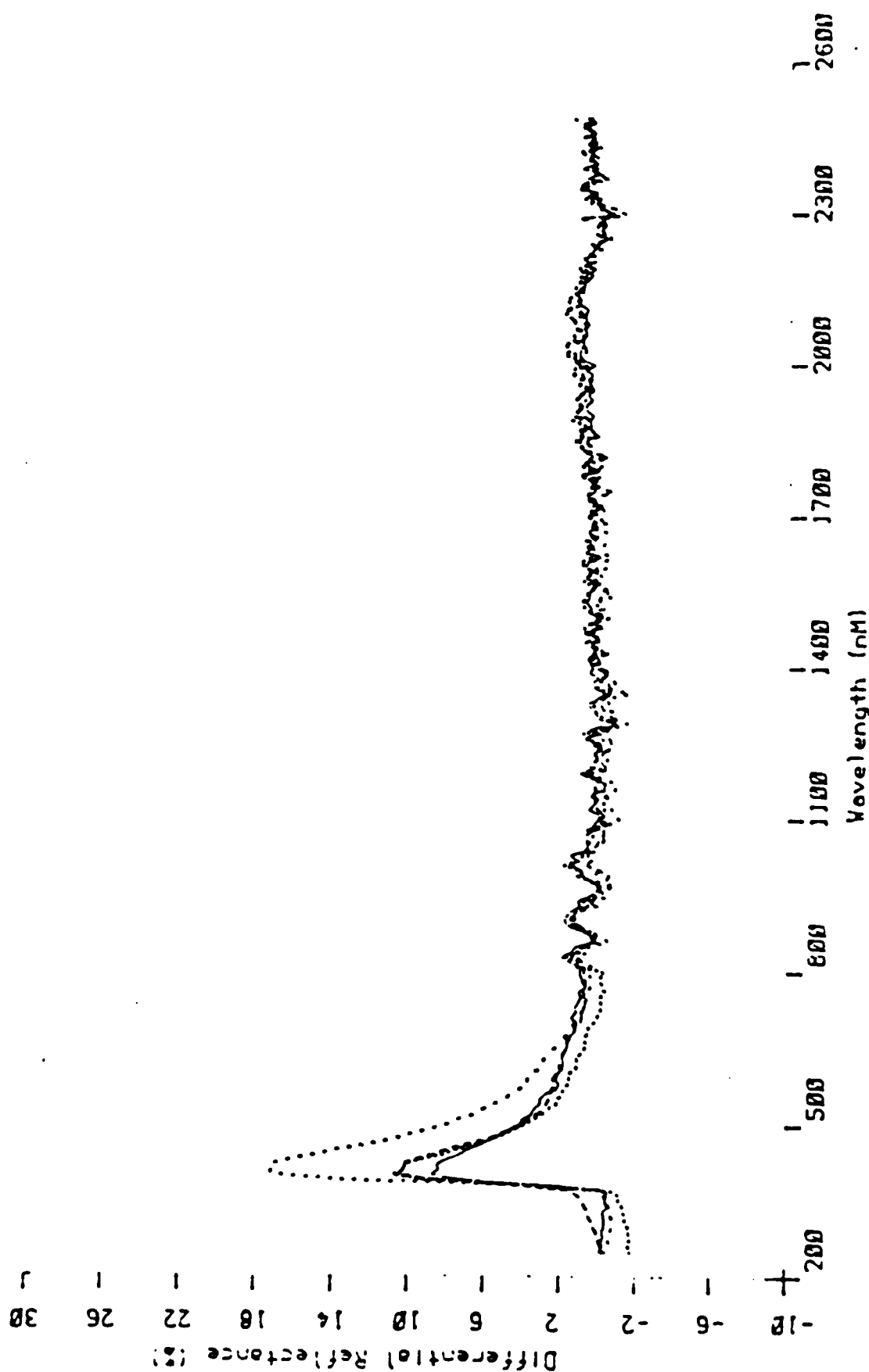


Figure 11.

# Alzak Performance

- : ■ J2 (2.5 (UVS)  
 ..... : ■ 5 (unexposed)

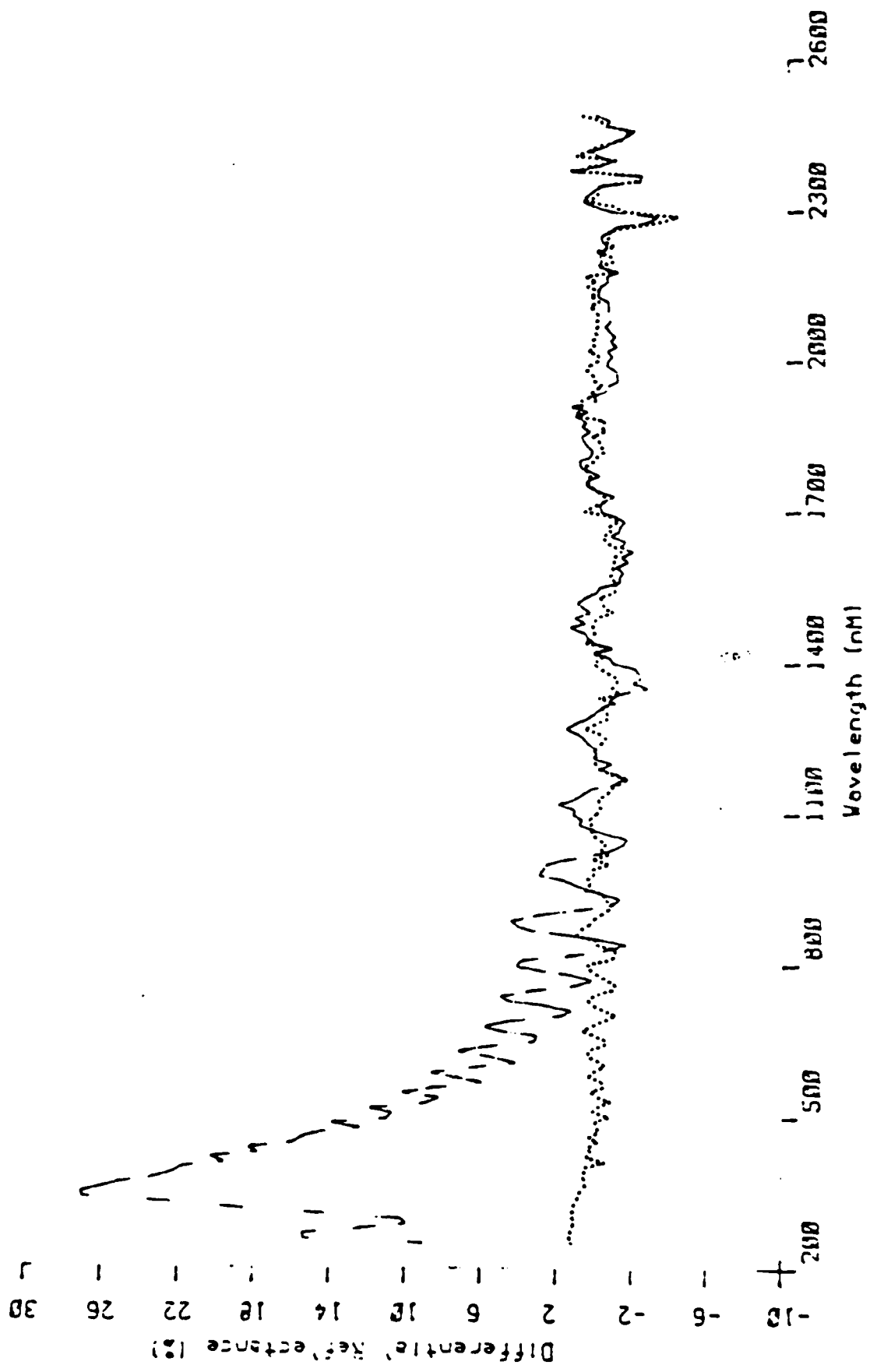


Figure 12

RTV - J2 recovery at 425 nM

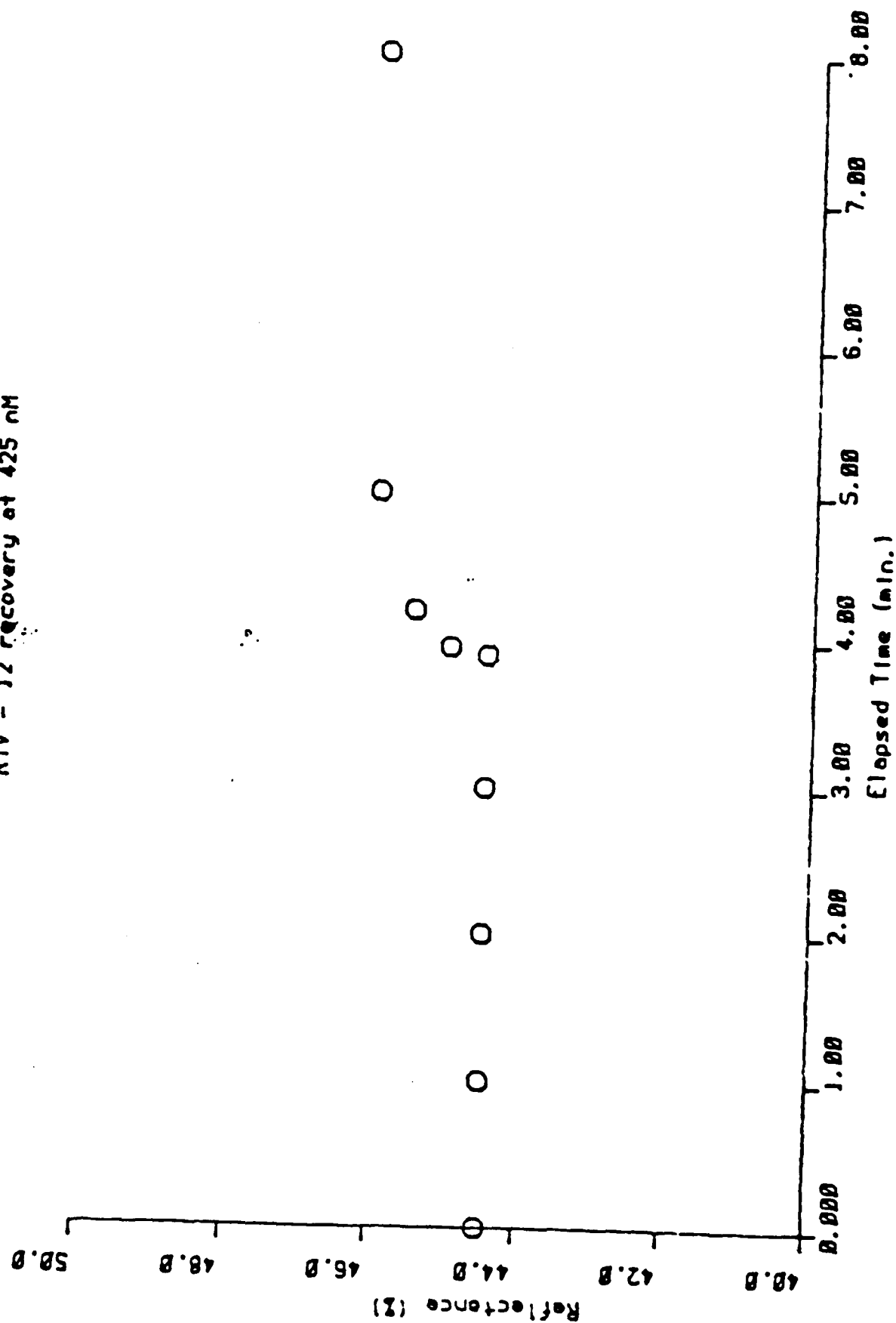


Figure 13

## PART II

### PROCEDURES AND SUPPORTING DATA

#### GENERAL SYSTEM PERFORMANCE

The run began with pre-test measurements of the test samples on both the DK-2A and M-IV spectrophotometers. The chamber was then cleaned and loaded, and pumpdown began in the first week of April. Chamber pressures in the  $10^{-7}$  torr range were reached after bakeout (a plot of chamber pressure versus time is presented in Figure 14) and testing progressed as planned, after initial adjustments of the electron guns had been completed.

Simulated solar/electron exposure began on 16 April.

Almost immediately, problems occurred. The facility mini-computer malfunctioned shortly after the 0 ESH spectrophotometer measurements were taken. In view of the urgent need for data on the S-13G/LO materials, it was decided to continue the test, taking the initial measurements by hand; until repair parts for the computer arrived.

An electrical problem on 19 April rendered both electron guns and the mass spectrometer inoperative. The high energy (8 KeV) gun was returned to operation on 23 April, but the low energy gun was found to have a damaged firing unit which could not be repaired without opening the system.

Exposure continued until 29 April, at which point it was decided to stop exposures until the computer was repaired. This was accomplished the following week, and exposure was started again on 9 May. Spectrophotometer measurements were made manually before this hold, and visual comparison with the data taken afterward indicated no change during the one-week vacuum soak. The test continued normally until 13 May, when the solar simulator developed electrical problems (probably as a result of the aforementioned brownout). During the following week, exposure was started and stopped four times - in each case the solar simulator malfunctioned. Eventually, the problem was isolated, and exposure continued without problems until the test concluded on 20 May.

## SPECTRO-RADIOMETER EXPERIMENT

### Description

The spectro-radiometric measurement equipment is a locally produced prism instrument, constructed from the monochromator of a defunct Beckman DK-2A spectrophotometer, combined with a U.V. sensitive silicon photodiode and locally-built amplifiers. Data is collected using the facility computer.

### Results

The computer problems mentioned above prevented use of the spectro-radiometer during this run. However, exposure was measured before and after the test using a Yellow Springs Instrument radiometer, during the test with an in-situ silicon photodiode, and experimentally using a photoelectric effect detector (see Electron Detector experiment). All three measurements agree to within .2 EUVS with the figures stated above, and indicate no change in exposure intensity during the test. This assumption is further supported by Figure 15 which presents temperature of the Alzak #12 specimen versus time. The temperature is nearly constant at 125°F, indicating constant exposure intensity with time.

Figure 16 presents a plot of the nominal ATSS spectrum with the solar spectrum overlaid for comparison purposes.

## ELECTRON DETECTOR

### Description

Two electron detectors are used in SCEPTRE to monitor the flux from the electron guns. The first is a modified Faraday detector using a conical geometry, and coated with gold to minimize secondary electron emission effects. The second is a simple disk of aluminum located on the sample wheel, which can be rotated to observe the flux at each sample position. Either detector may be read out using a Keithly Model 414 nanoammeter.

### Results

Unfortunately, the cone detector could not be used during this run due to installation problems. In view of the urgency of this test, it was decided to use only the aluminum disk detector.

The disk detector has a severe problem caused by secondary electron emission at low energies. This is presented in Figure 17 where electron current from the detector is plotted as a function of accelerating voltage (the emission current from the gun was constant). The "dip" in the curve at ~300 volts is due to secondary emission from the detector. However, at higher energies (5-15 KeV), the detector works reasonably well.

Accordingly, the procedure used was to set up and adjust each of the two guns at 8 KeV, and then to readjust the low energy gun to 400 volts. Since the guns are designed to maintain constant emission regardless of voltage, this permits a reasonably simple and accurate setting. However, as noted above, the low energy gun failed after only two days operation, which eliminated this problem.

From this point on, the single operating gun was operated at 8 KeV, with source current adjusted to yield 8 nA on the disk at the top dead center sample position (2.5 EUVS). The disk is 15/16" in diameter, so a simple computation yields the flux:

$$F = I / (A * C)$$

where

F = flux (e-/cm<sup>2</sup>/sec)

I = detector current (amps=coulombs/sec)

A = detector area (cm<sup>2</sup>)

C = electron charge (coulombs)

Which works out to  $12 \times 10^9$  e-/cm<sup>2</sup>/sec. Currents in the other operating positions were 6 nA in the 2 EUVS positions, 4 nA in the 1 EUVS positions; which converts to electron fluxes of  $9 \times 10^9$  and  $6 \times 10^9$  e-/cm<sup>2</sup>-sec, respectively. These levels were constant throughout the test.

## PHOTOELECTRIC EFFECT MEASUREMENT

An additional use for the disk detector was found on 17 May 85, when the operator discovered that a .45 nA current was present in the detector with the electron gun off. Blocking the solar simulator beam eliminated the current. A quick experiment was then conducted - the detector was stimulated with U.V. photons from a "mining light" (a low-pressure mercury arc lamp), and a small but measurable, current was detected. In both cases it was found that interposition of a pyrex filter eliminated the current (pyrex is opaque at U.V. wavelengths).

This ultraviolet induced current may be explained by invoking the Einstein photoelectric effect, in which electrons are ejected from a material by the arrival of energetic photons. Rough calibration of the disk for this purpose was attempted, using a deuterium lamp as a calibration source, but the deuterium lamp produced currents too low to read out. However, this still permitted an order of magnitude calculation to be made, as follows:

Work function for Al = 4.1 eV

Conversion factor = 1240 eV-nm

Therefore, minimum excitation wavelength =  $1240/4.1 = 302$  nm  
(any photons of lower energy have no effect)

Response of in-situ silicon photodiode to deuterium lamp:  
.64 uA (through sapphire window)  
.40 uA (through sapphire and pyrex)

Therefore, response at U.V. wavelengths =  $.64 - .4 = .24$  uA  
(pyrex is opaque at U.V. wavelengths below 310 nm)

Responsivity = .08 A/W (from manufacturer's specs)

Therefore, optical power =  $.24/.08 = 3$  uW

Correction for 80% transmission of sapphire window:  
 $3 \text{ uW}/.8 = 3.75 \text{ uW}$

Area of detector =  $.3841 \text{ cm}^2$

Therefore, U.V. intensity =  $3.75 \text{ uW}/.3841 \text{ cm}^2 =$   
 $1.08 \times 10^{-5} \text{ W/cm}^2$

However, the disk current is unreadably low ( $I < .001 \text{ nA}$ )  
for this source

Therefore, given disk area =  $4.45 \text{ cm}^2$

Disk sensitivity  $S_d < .001 \times 10^{-9} \text{ A} / (1.08 \times 10^{-5} \text{ W/cm}^2 \times 4.45 \text{ cm}^2)$

Therefore,  $S_d < 2.1 \times 10^{-8} \text{ A/W}$

Disk yields  $.45 \times 10^{-9} \text{ A}$  for ATSS

Therefore,  $I_{atss} > .45 \times 10^{-9} \text{ A} / (2.1 \times 10^{-8} \text{ A/W} \times 4.45 \text{ cm}^2)$   
 $> 4.82 \times 10^{-3} \text{ W/cm}^2$  at wavelengths  $< 302 \text{ nm}$

From Thekakra's solar spectrum data, the sun has  $1.9 \text{ mW/cm}^2$  at wavelengths  $< 302 \text{ nm}$

So,  $I_{atss}/I_{\text{sun}} > 4.82 \text{ mW/cm}^2 / 1.9 \text{ mW/cm}^2 = 2.54 \text{ EUVS}$

The extremely good agreement with the 2.5 EUVS value is probably fortuitous, but it's certainly encouraging.

## SPECTROPHOTOMETER EXPERIMENT

### Description

A Beckman ACTA M-IV spectrophotometer is used to measure the reflectance of test specimens inside the vacuum chamber. It is a fairly typical grating-type spectrophotometer, as used by hospitals, with certain modifications:

1. In-situ integrating sphere and detectors
2. Xenon arc lamp for U.V. - visible illumination
3. Computer interface for digital data storage.

The M-IV is a double-beam ratio recording instrument with all solid state electronics, and is capable of producing data in the range  $160 \text{ nm} - 2600 \text{ nm}$  in our installation. This range is reduced to  $250 \text{ nm} - 2500 \text{ nm}$  because of the limitations of the older DK-2A instrument used as a reference. The data is generally accurate to  $\pm 2.0\%$ , and has a high degree of repeatability provided that great care is taken by the operator to do everything in exactly the same way from test to test.

Computation of solar absorptance from the corrected reflectance data is performed using the University of Dayton's MIST (Macro Interpretive Spectral Translator) software. This method differs from standard hand calculation in the ability to use a much larger number of points - each solar absorptance datum is based on 225 reflectance values each of which is itself an average over three successive measurements. As a result, absorptances

computed using this technique are considered accurate to  $\pm .005$ , when compared with other measurements performed in the same manner on the same instrument. As can be seen by comparing the DK-2 and M-IV columns in Table 2, comparison with other instruments is difficult.

### Results

Figures 1-5 and 18-20 present pre- and post-test reflectances, in air and in vacuum, for all specimens, including the four which were shielded. Two points about these charts are worthy of note.

1. Figures 5 and 20 present reflectances of the two Alzak specimens in this test. Two previously unreported effects are noted - a 2% drop in reflectance on pumpdown, and recovery of the damaged specimen on exposure to air.

2. Figures 18 and 19 present reflectances of two S-13G/LO specimens which were exposed to vacuum, but not to ionizing radiation. A nearly uniform 1% drop in reflectance is noted. Visually, these samples presented a glossy appearance when inserted in the system, and a matte appearance when removed. This suggests that excess binder may have been removed by the bakeout (72 hours at  $\sim 185^{\circ}\text{F}$ ).

Because of Items 1 and 2 above, it was decided to examine the specimens for cross-contamination using an FTIR spectrometer. No common absorption bands were found for the Alzak and S-13G/LO specimens (indeed, the exposed Alzak showed no new absorption bands at all, which suggests that the damage mechanism is atomic, rather than chemical), and on this basis, it is considered unlikely that cross-contamination occurred. Precisely why the unexposed Alzak dropped in reflectance is, therefore, unknown. However, it should be noted that the bakeout in this test was conducted for longer, and at higher temperatures, than in previous tests. One possibility is that separation of the anodized coating from the aluminum substrate occurs due to thermal expansion.

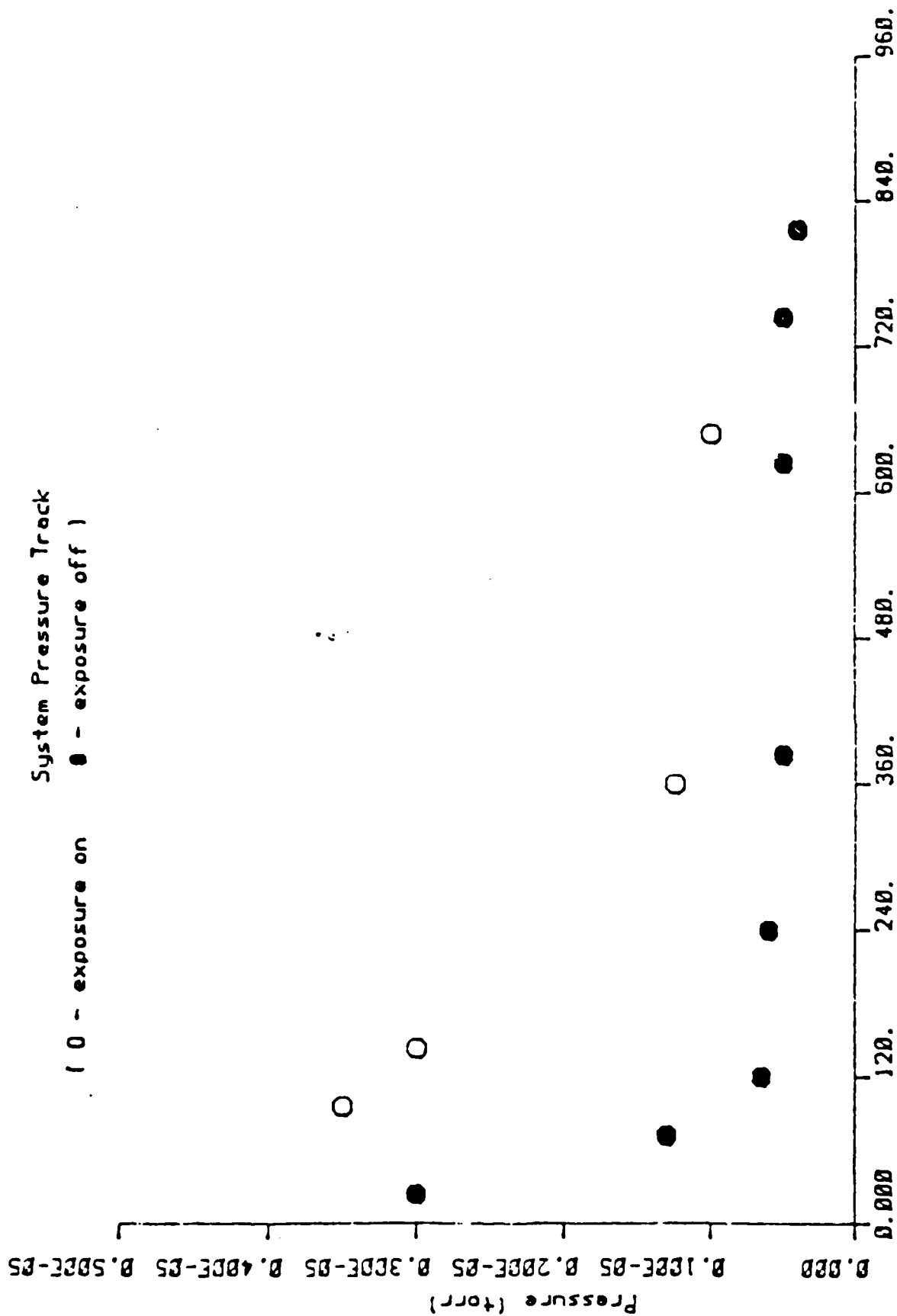


Figure 14

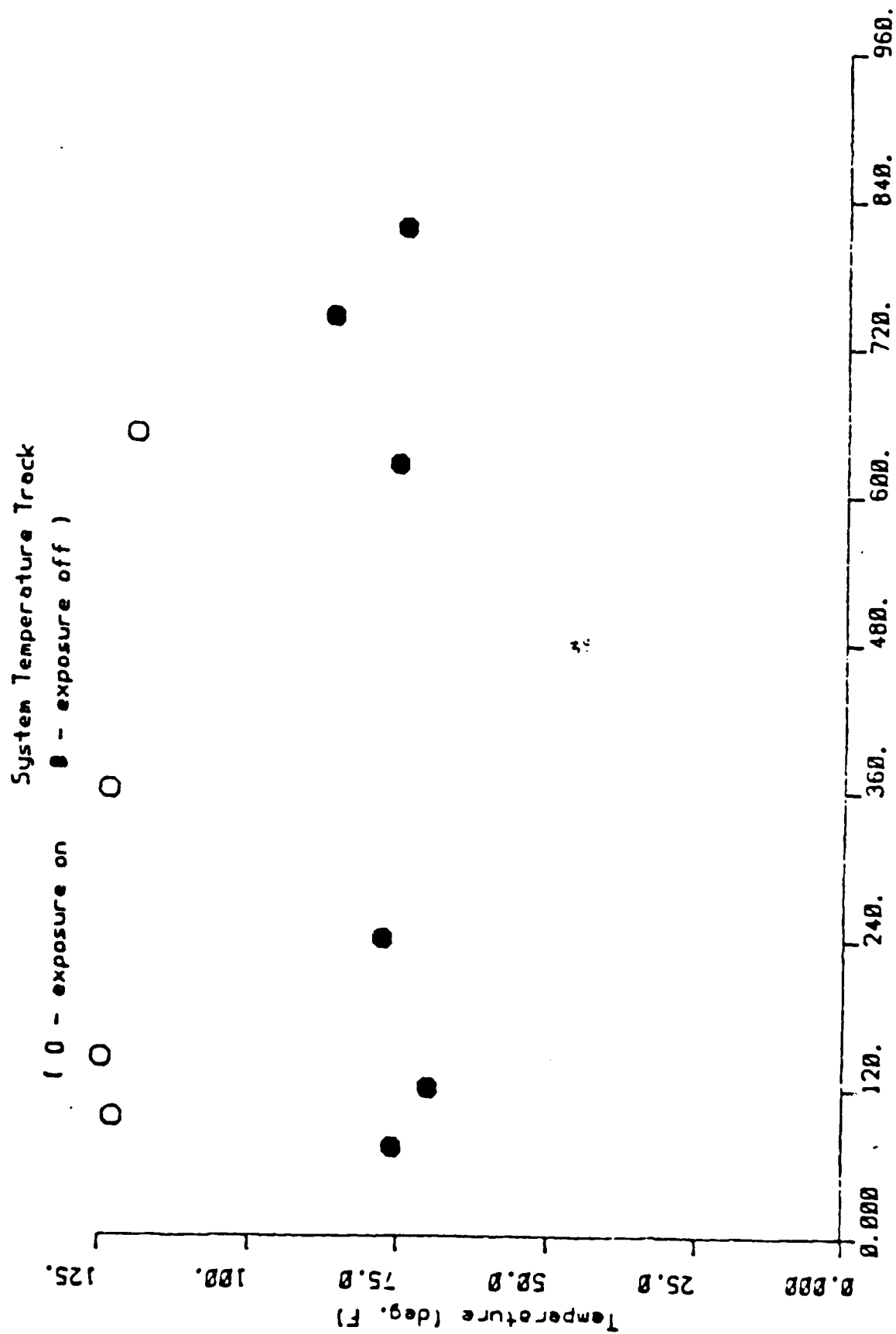


Figure 15

# Spectra of Sources

- : Sun (air mass 0)  
 ..... : ATSS (estimated)

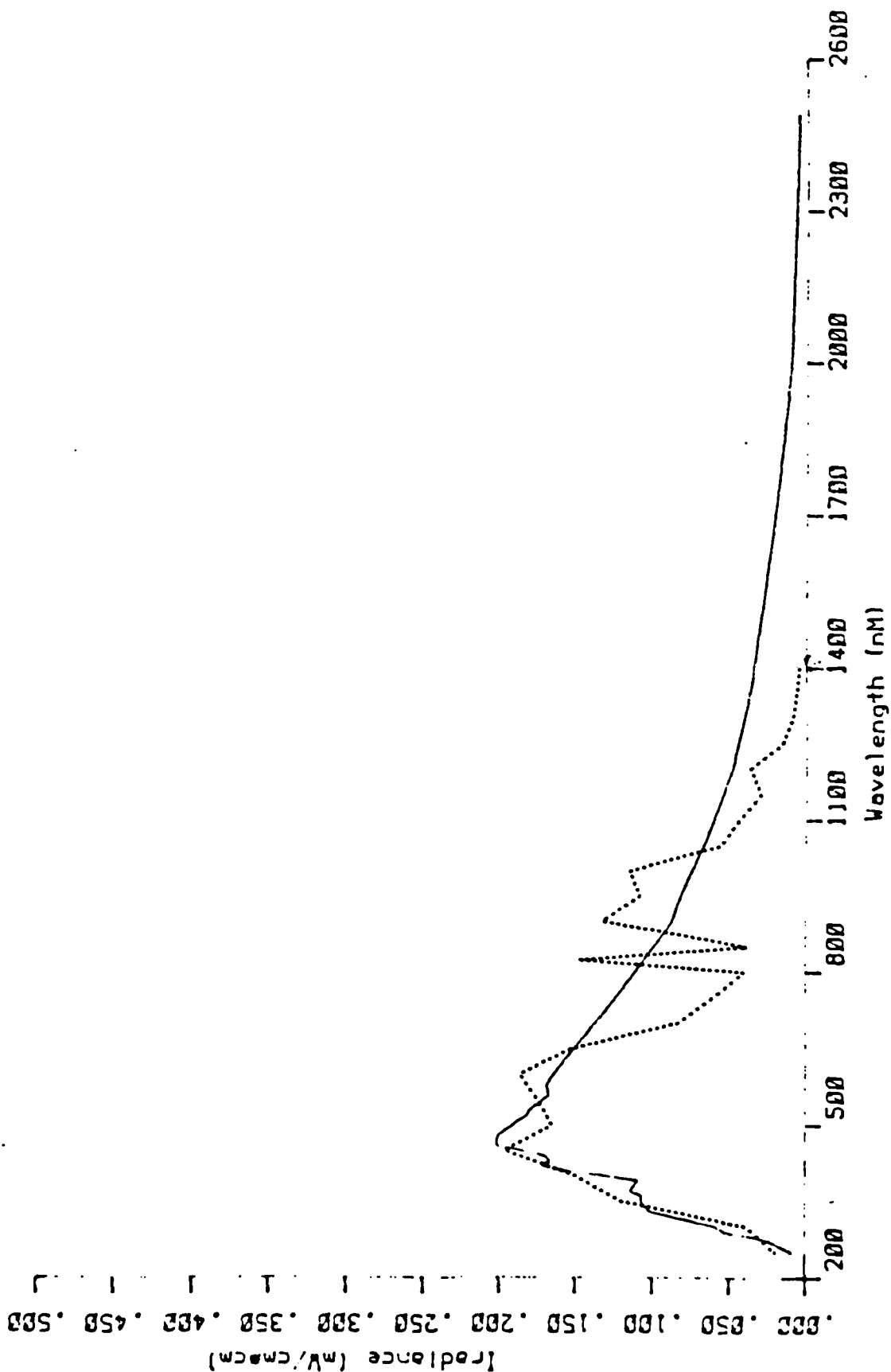
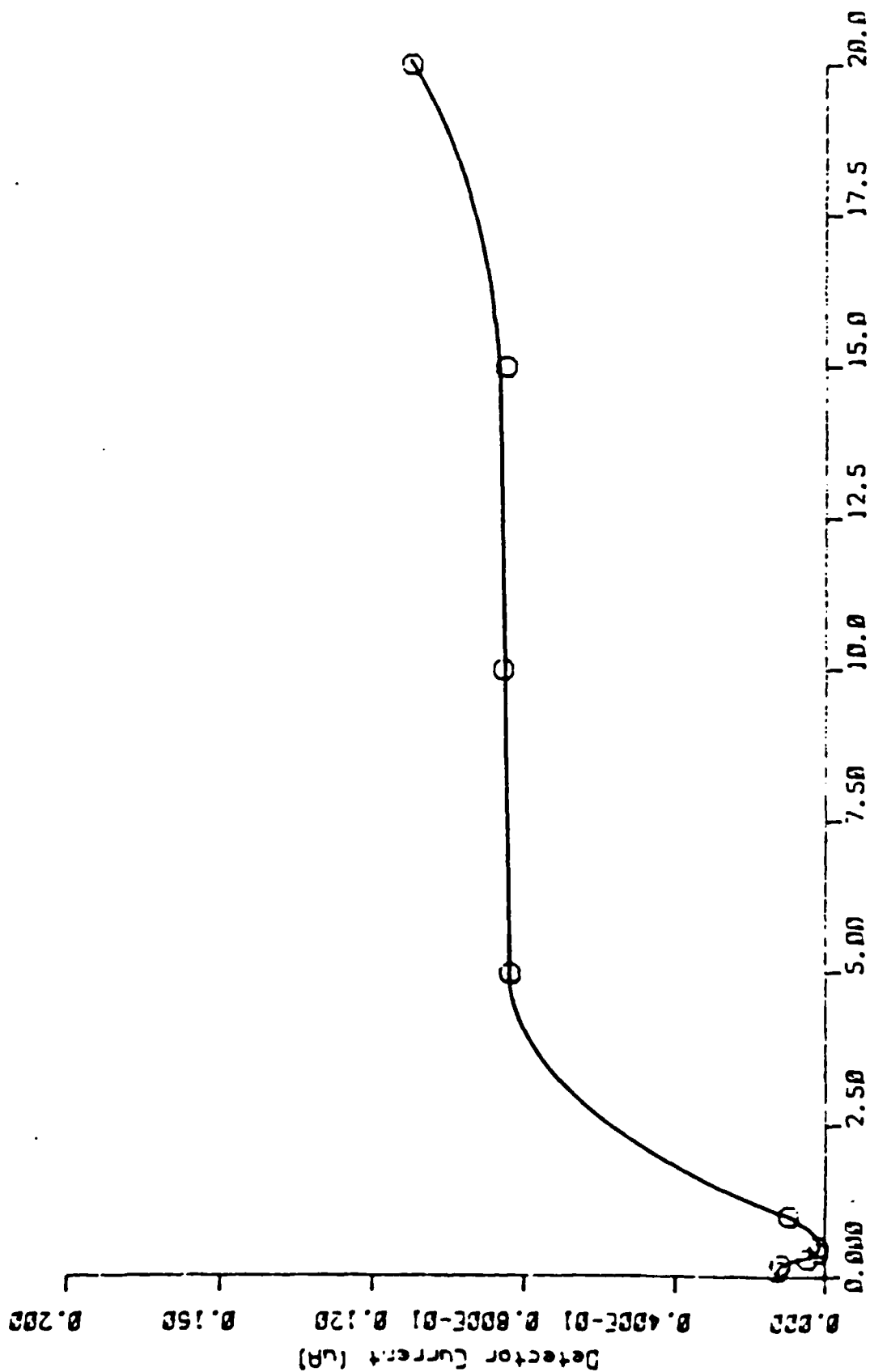


Figure 16

# Electron Detector Response



Energy (KeV)

Figure 17

S-13 • 3

..... : pre - test (air)  
..... : pre - test (vac)  
..... : post - test (vac)  
..... : post - test (air)

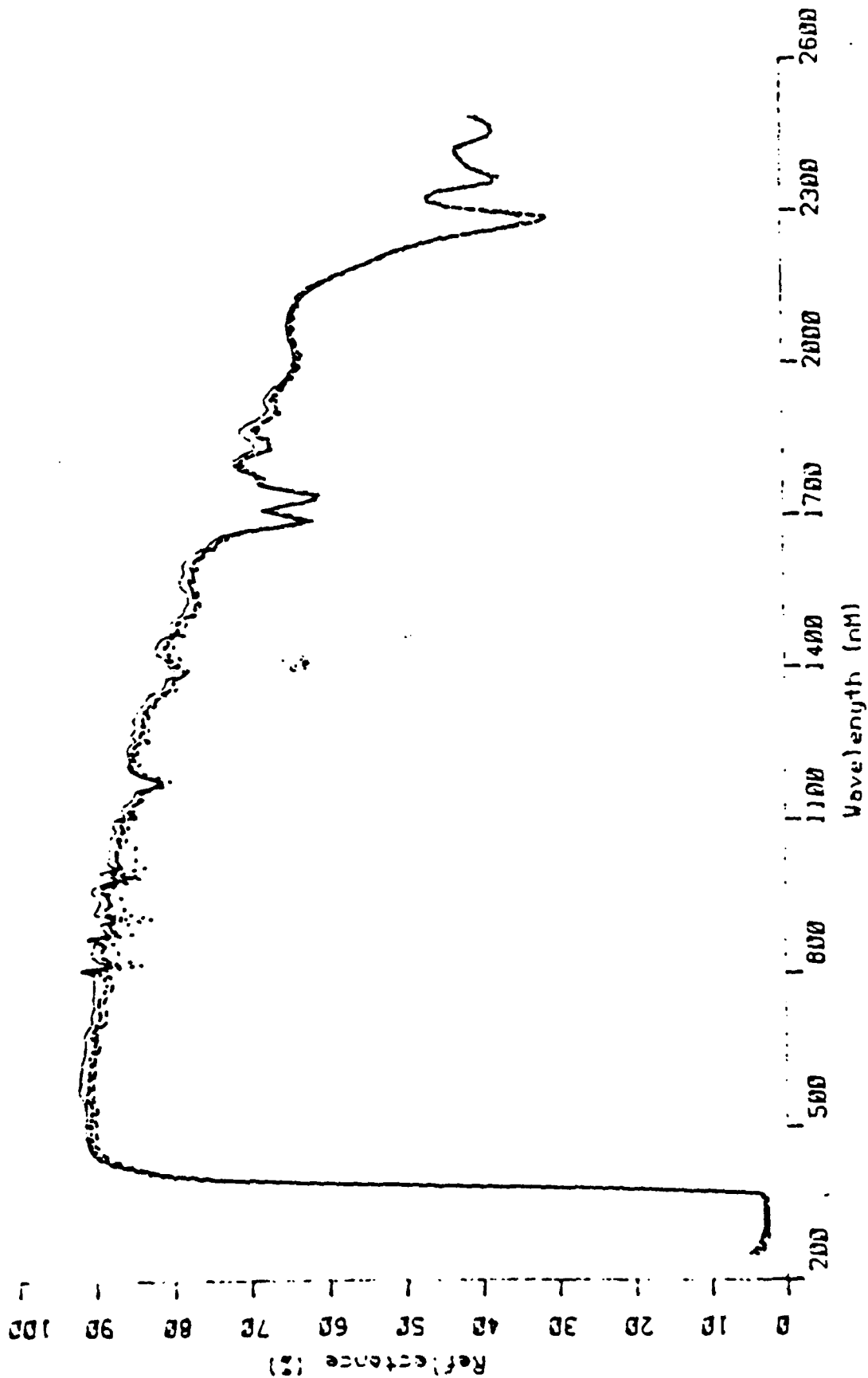


Figure 18

S-13 0 4

- : pre - test (air)  
..... : pre - test (vac)  
..... : post - test (vac)  
..... : post - test (air)

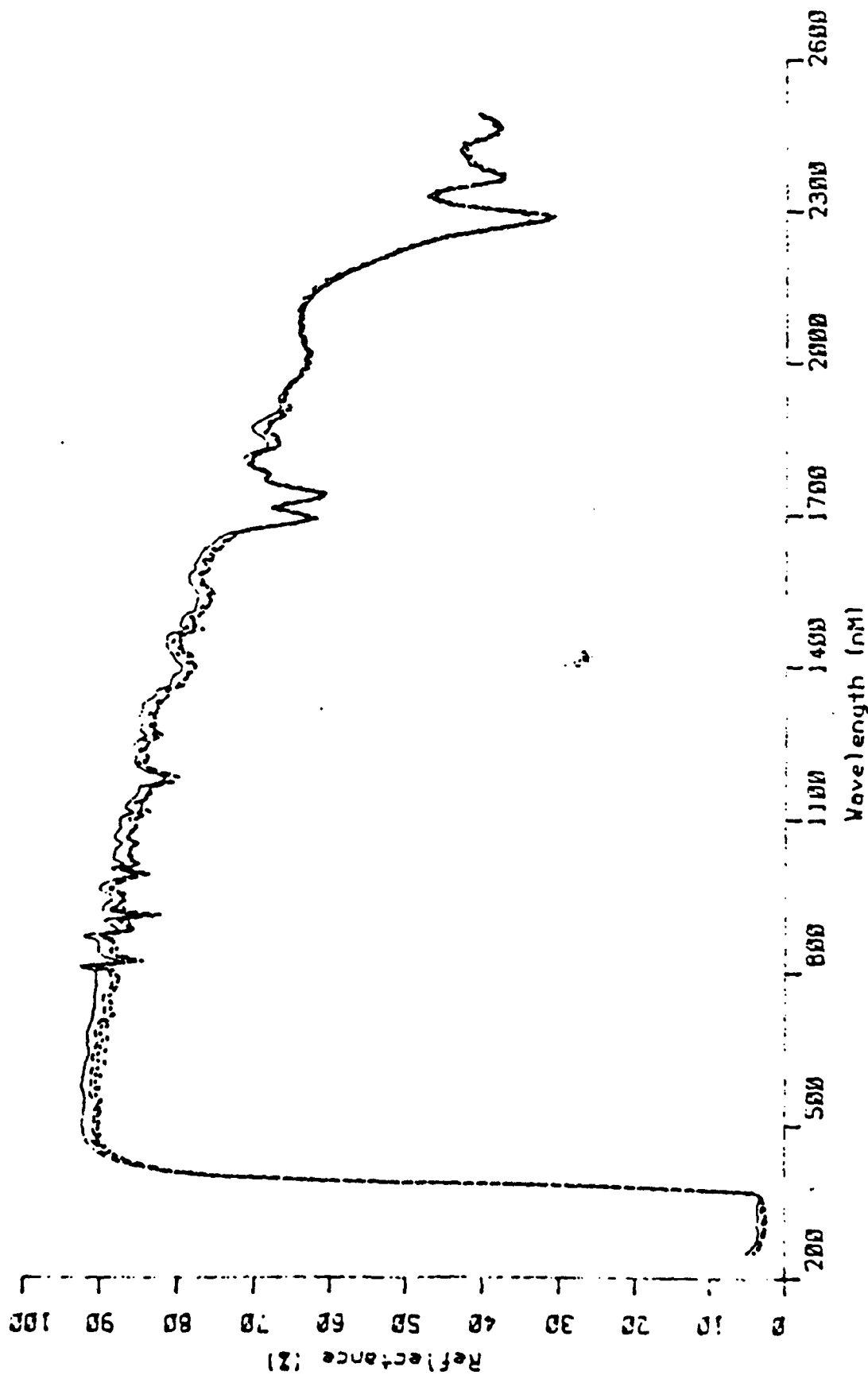


Figure 19

Hizok # 9

- : pre - test (air)  
 ..... : pre - test (vac)  
 ..... : post - test (vac)  
 ..... : post - test (air)

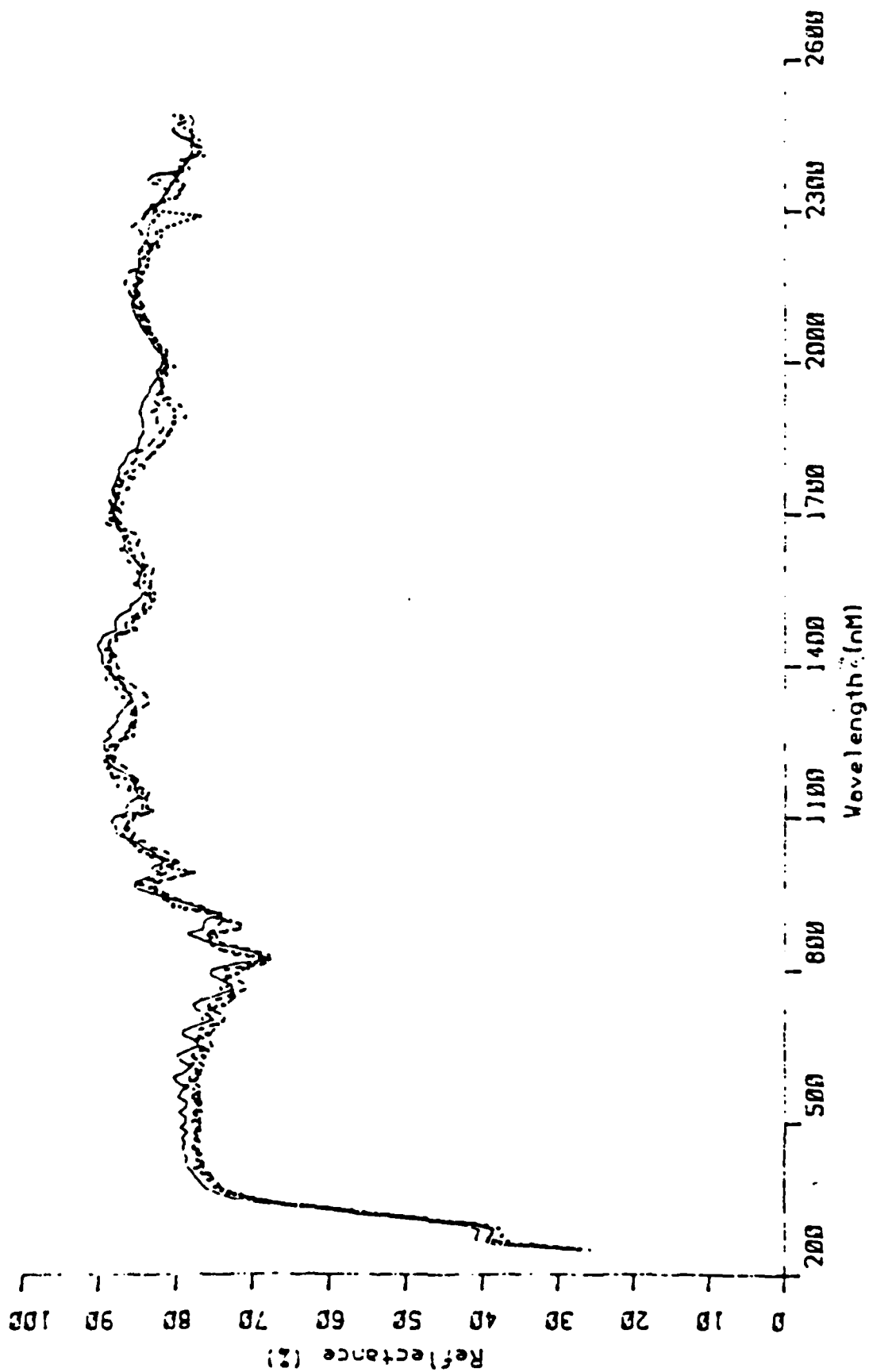


Figure 20

**APPENDIX D**  
**DISTRIBUTION LIST AND SAMPLE LETTER FOR S13G/L0-1**

DISTRIBUTION LIST FOR S13G/L0-1

AEROJET ELECTROSYSTEMS CO.

Daryl L. Mossman  
1100 W. Hollyvale Street  
P.O. Box 296  
Azusa, CA 91702

AEROSPACE CORPORATION

Larry Rachal, M.S. M4-967  
P.O. Box 92957  
Los Angeles, CA 90009

Gene Borson, M.S. M2-250

BOEING AEROSPACE CO.

Sylvester G. Hill, M.S. 87-63  
P.O. Box 3999  
Seattle, WA 98124

Larry Fogdall, M.S. 2R-00  
P.O. Box 3707

FORD AEROSPACE & COMMUNICATIONS

John Hamachi, M.S. G97  
3939 Fabian Way  
Palo Alto, CA 94303

GENERAL ELECTRIC

Dr. A. T. Tweedie  
Space Systems Div.  
Valley Forge Space Center  
P.O. Box 8555  
Philadelphia, PA 19101

HUGHES AIRCRAFT CO.

Robert Champetier  
2000 El. Segundo Blvd.  
Bldg. E54/F220  
El Segundo, CA 90245

David Williams  
2000 N. Sepulveda  
Bldg. E-12/M.S. A120

LOCKHEED MISSILES & SPACE CO.

Brian Petrie  
Org. 62-92, Bldg 564  
P.O. Box 504  
Sunnyvale, CA 94086

LOCKHEED MISSILES & SPACE CO.

Matt McCargo  
Dept. 52-32, Bldg. 205  
3251 Hanover St.  
Palo Alto, CA 94304

RCA ASTROELECTRONICS

Terri Cardellino  
Rts 535 & 571  
Princeton, N.J. 08540

ROCKWELL INTERNATIONAL  
Jack Olson/Mail Code AD 70  
Transportation Systems Div.  
12214 Lakewood Blvd.  
Downey, CA 90241

ROCKWELL INTERNATIONAL  
George Frey/Mail Code SL 48  
Satellite Systems Div.  
2600 Westminster Blvd.  
P.O. Box 3644  
Seal Beach, CA 90740-7644

TRW  
James Thomasson, M.S. 01-2220  
One Space Park  
Redondo Beach, CA 90278

TELEDYNE SYSTEMS CO.  
Ed Abrams, M.S. 70  
19601 Nordhoff Street  
Northridge, CA 91324

LTV AEROSPACE & DEFENSE CO.  
Roy L. Cox, M.S. EM-53  
Vought Missile & Advanced Programs Div.  
P.O. Box 650003  
Dallas, TX 75265-003

MARTIN-MARIETTA CORP.  
Martin Bolt, M.S. M0487  
P.O. Box 179  
Denver, CO 80201

NASA-AMES RESEARCH CENTER  
Dan Leiser, M.S. 234-1  
Moffet Field, CA 94035

NASA-GODDARD SPACE FLIGHT CENTER  
Ben Seidenberg, Code 732  
Greenbelt, MD 20771

NASA/JOHNSON SPACE CENTER  
Dr. L. J. Leger, Mail Code ES53  
NASA Road One  
Houston, TX 77058

NASA-LANGLEY RESEARCH CENTER  
Wayne Slomp, M.S. 183  
Bldg 1256  
Hampton, VA 23665

NASA-MARSHALL SPACE FLIGHT CENTER  
Roger Harwell/EH 34  
Marshall Space Flight Center, AL 35812

NASA-LEWIS RESEARCH CENTER  
James P. Couch, M.S. 500-107  
21000 Brookpark Road  
Cleveland, OH 44135

JET PROPULSION LABORATORY  
Robert Hale, M.S. 157-102  
4800 Oak Grove Drive  
Pasadena, CA 91109

SANDIA NATIONAL LABORATORY  
Douglas Salmi, M.S. 74  
Division 5332  
P.O. Box 5800  
Albuquerque, NM 87185

NAVAL RESEARCH LABORATORY  
Nelson Hyman, Code 7734  
Washington, D.C. 20375-5000

HARRIS CORPORATION  
Jeff Hooper  
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P.O. Box 9400  
Melbourne, FL 32902

HONEYWELL, INC.  
Bill Mosker  
Aerospace Division  
13350 U.S. Hwy 19 South  
Clearwater, FL 33546



IIT Research Institute  
10 West 35th Street, Chicago, Illinois 60616-3799  
312/567-4000 • Telex 282472 IITRICHGO

## SAMPLE LETTER

October 24, 1986

Jule Hirschfield  
NASA/Goddard Space Flight Center  
Mail Code 754  
Greenbelt, MD 20771

Dear Mr. Hirschfield:

Because of your interest in spacecraft thermal control, you are aware that IITRI's spacecraft thermal control coating, S13G/L0, is in the process of being phased out. In order to provide a continuing supply of this stable elastomeric coating, we have conducted a program for the Air Force under Space Division funding to develop and qualify a material having the same or improved optical and physical properties as S13G/L0. We believe we have achieved this goal.

As part of the completion of this program, "Space Stable Thermal Control Coatings", Contract F33615-83K-5099, managed by the Air Force Wright Aeronautical Laboratories/Materials Laboratory, we have formulated a production batch of this new material designated S13G/L0-1. We would be pleased to have you join in the evaluation of this material since you have shown both an interest and expertise in working with thermal control coatings in the past. A one pint quantity can be supplied at no cost to you or some other interested person in your organization. Your request should be directed to:

Yoshiro Harada or Richard J. Mell  
IIT Research Institute  
10 West 35th Street  
Chicago, Illinois 60616  
312/567-4432

Storage, handling, and processing instructions will be included with the material. Please indicate a mailing address which will expedite air express delivery of the S13G/L0-1. Please have your request in to us as soon as possible upon receipt of this letter. Our only request is a willingness to openly share the results of your evaluation along with your assessment as soon as is feasible.

We hope that you will find S13G/L0-1 to be a good successor to S13G/L0. The indications from NASA-Marshall on their qualification tests are quite favorable.

Thank you for your interest and cooperation.

Sincerely,

  
[Redacted]  
Senior Engineer  
Chemical Analysis Section

YH/cae

END

8-87

DTIC